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INDEX NUMBER

CONTAINING

TITLE-PAGE, INDEX, &c., TO VOLUME LXXVI.

CONTENTS.

ARTICLES:—	PAGE
On the Refractivities of Air, Oxygen, Nitrogen, Argon, Hydrogen, and Helium, by Prof. William Ramsay, Ph.D., LL.D., Sc.D., F.R.S., and Morris W. Travers, B.Sc.	1
Hysterical Chemistry, by Prof. H. Carrington Bolton, Ph.D. ..	3
Laboratory Notes—Asbestos, Combustion Furnaces, by H. Jarvis	5
PROCEEDINGS OF SOCIETIES—	
CHEMICAL SOCIETY.—Stereochemistry of Unsaturated Compounds. Part I. Esterification of Substituted Acrylic Acids—Formation and Hydrolysis of Esters—A New Method of Determining Freezing-points in very Dilute Solutions	7
CHEMICAL NOTICES FROM FOREIGN SOURCES	10
MISCELLANEOUS	12

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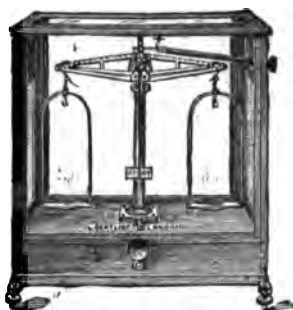
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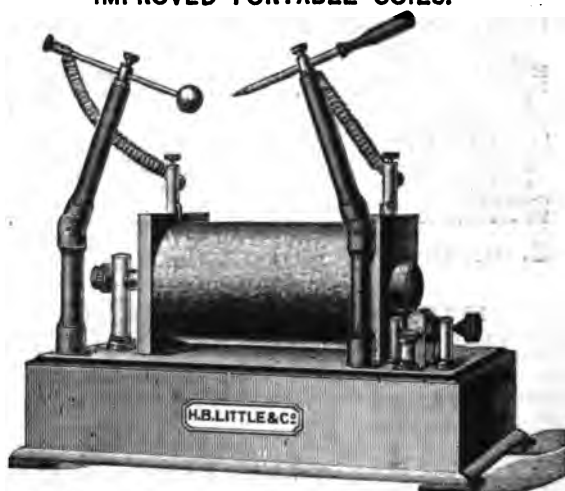
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THE CHEMICAL NEWS.

VOLUME LXXVII.

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No. 1989.—JANUARY 7, 1898.

ON THE REFRACTIVITIES OF AIR, OXYGEN, NITROGEN, ARGON, HYDROGEN, AND HELIUM.*

By Prof. WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S.,
and MORRIS W. TRAVERS, B.Sc.

IN the course of a research on the nature of helium many measurements of its refractivity referred to that of air as unity were made by means of an apparatus similar to that described by Lord Rayleigh (*Proceedings*, vol. lix., p. 203). Inasmuch as the refractivity of helium is very small, it was not found convenient to measure its value directly against air; hence it was compared with hydrogen, and hydrogen was compared with air. And as a check on these measurements, the hydrogen was compared with oxygen, and subsequently with nitrogen free from argon. It was noticed, after some of these experiments had been made, that the refractivity of air could not be accurately calculated from the given data for oxygen, nitrogen, and argon; and it appeared therefore worth while to examine more minutely the refractivity of these gases for white light, and to see whether any error could be detected in previous measurements. Moreover, as physicists perhaps do not always devote sufficient care to the chemical purity of their materials, an additional reason was furnished for the inquiry.

Apparatus.—It will be seen, on consulting Lord Rayleigh's paper, that the refractivity is measured in the following manner:—Light from a paraffin lamp passes through a fine slit, cut with a razor in tin-foil pasted on glass. The beam is made parallel by passage through an achromatic plano-convex lens of about 1 foot focal length. It then divides; the upper portion passes through air, and, after extraneous light is cut off by passage through two wide slits, it is brought to a focus by a lens similar to the first, and the bands produced are viewed by a cylindrical lens of very short focus. The lower portion of the beam traverses two tubes, 9 inches long and $\frac{1}{4}$ of an inch in diameter, placed close together, and closed at each end with plates of optically worked glass. Each of these tubes contains one of the gases to be examined, and each is connected with a manometer and a movable reservoir; so that, on raising or lowering the reservoir, the pressure of the gases can be so adjusted that the interference-bands formed in the lower half of the field can be accu-

ately brought into line with the stationary bands in the upper half. Readings of pressure are taken on both manometers at pressures not differing greatly from that of the atmosphere; then, on lowering the reservoirs, readings on both manometers are taken at lower pressures, the bands being again made to coincide in position with the upper fiducial bands. The ratio of the refractivities is inversely as the differences of pressure in the two gases. The influence of temperature does not appear, for the tubes of the manometer lie side by side, and may be regarded as equally affected by variations of temperature.

The accuracy of this method varies with the value of the refractivity of the gas. For if the gas has a low refractivity, then a great difference of pressure produces the passage of fewer bands across the field than if it has a high one; and, as the accuracy of reading may safely be taken as the twenty-fifth of a band; and as between thirty and forty bands passed the field with such gases as oxygen, nitrogen, and argon, the error may be taken in such cases as from 1 in 750 to 1 in 1000.

The tubes containing the gases to be examined were connected with a Töpler's pump; and before admission of gas each tube was pumped empty, so that in an attached Plücker's tube there was brilliant phosphorescence. The tubes were then washed out with the gases to be admitted, the apparatus again evacuated, and the final quantity of gas allowed to enter by a contrivance a description of which is to be found in the *Trans. Chem. Soc.*, vol. lxvii., p. 686.

Purity of the Gases. Hydrogen.—The hydrogen was made by warming a tube containing palladium hydrogen which had been prepared by admitting hydrogen made from pure zinc and sulphuric acid into contact with spongy palladium. The tube was pumped empty in the cold, and then gently warmed; it was again allowed to cool, and again pumped empty. The hydrogen was then collected passing slowly through a tube, filled with phosphoric anhydride, into the experimental tube.

Oxygen.—The oxygen was prepared by heating a small tube containing potassium permanganate; a large quantity of gas was allowed to escape, and a portion was collected finally which served for the experiments.

Nitrogen.—The nitrogen was prepared from a mixture of ammonium chloride and sodium nitrite, to which a little copper sulphate had been added. The apparatus was exhausted before admission of either of the solutions, and before allowing the solutions to enter they were boiled, and the flasks corked while boiling. The gas was

* A Paper read before the Royal Society, December 9th, 1897.

passed over red-hot copper; the ammonia liberated by the alkalinity of the nitrite thus reacted with any oxides of nitrogen possibly present to form water. The gas was collected, after rejection of a considerable portion, in a tube containing oil of vitriol; it was then transferred to a fresh tube, treated with a very strong solution of caustic potash, and finally admitted to the apparatus.

Air.—The air was left standing for some hours in a tube containing sticks of caustic potash, and was then admitted to the apparatus through a tube of phosphoric anhydride.

Experimental Data.—Each gas was compared with air and with the other two. Air is in each case taken as unity.

Hydrogen. —Hydrogen/air		0.4730	Mean 0.4733
		0.4737	
Hydrogen/oxygen		0.5125	
		0.5125	
Hydrogen/nitrogen		0.4654	" 0.4654
		0.4654	
 Oxygen. —Oxygen/air		0.9237	" 0.9243
		0.9262	
		0.9230	
Oxygen/hydrogen		1.9512	" 1.9512
		1.9512	
Oxygen/nitrogen		0.9090	" 0.9105
		0.9122	
		0.9103	
 Nitrogen. —Nitrogen/air		1.0153	" 1.0166
		1.0171	
		1.0174	
Nitrogen/hydrogen		2.1487	" 2.1487
		2.1487	
Nitrogen/oxygen		1.1001	" 1.0983
		1.0962	
		1.0986	

To these numbers those for argon must be added. The gas was prepared in the usual manner from air; and before admitting it into the experimental tube it was sparked with oxygen in presence of caustic soda for two days. The oxygen was removed with phosphorus, and the argon, on its way into the experimental tube, passed over phosphorus pentoxide; a Plücker's tube was sealed to the tube through which it entered, so that its spectrum might be observed. It contained no visible trace of either hydrogen or nitrogen.

Argon. —Argon/air		0.9596	Mean 0.9596
		0.9596	
		0.9598	
Argon/oxygen		1.0350	
		1.0348	" 1.0362
		1.0381	
Argon/nitrogen		0.9412	" 0.9416
		0.9419	

Placing air in each case equal to unity, and calculating the refractivities of the other gases, we obtain the following table:—

Refractivities of Gases, Air equal to Unity.

	Directly compared.	Through			
		Oxygen.	Nitrogen.	Hydrogen.	Argon.
Hydrogen	0.4733	0.4737	0.4727	—	—
Oxygen ..	0.9243	—	0.9247	0.9237	0.9261
Nitrogen ..	0.0163	1.0155	—	1.0170	1.0191
Argon ..	0.9596	0.9577	0.9572	—	—
Carbon dioxide	—	1.5316	—	—	—

It will be seen, on inspecting the above table, that the numbers obtained indirectly are in close agreement with those obtained by direct comparison with air.

Taking the value found directly by Mascart for D,* viz., $(n-1)_D = 0.0002923$, the values found by him for nitrogen (atmospheric) was 0.0002972, giving for nitrogen on the basis air equal to unity, the number 1.0178. Mascart did not determine the value for oxygen, but calculated it from the above data and the known composition of air. Nor did Lorenz determine the value for nitrogen, but, taking his own value for oxygen, viz., $(n-1)_D = 0.000272$, and for air $(n-1)_D = 0.000291$, he deduced it, as Mascart had done for oxygen. So that we have no determination of the three constants, or their comparison, by any one observer since Dulong in 1826. It has been tacitly assumed that the refractive index for a mixture of gases is that of those of their constituents, taken in the proportion in which they occur. We have in our hands a means of verifying this assumption, which is well known not to hold for compound gases, nor for mixtures of liquids, even though change of density be taken into consideration.

Dulong (*Ann. Chim. Phys.*, lxxxi., p. 176, 1826) gives very careful accounts of the methods he used in preparing the samples of gas that he employed. Oxygen, to which he ascribed the refractivity 0.924, was obtained by heating potassium chlorate. His result is identical with ours. Nitrogen was prepared from air by absorbing the oxygen with phosphorus, first at a high temperature and then in the cold. It was then washed with a solution of chlorine, and afterwards with potash. It is difficult to see what object was to be gained by washing with chlorine water, unless it was the removal of hydrogen. The number he obtained was 1.02, somewhat higher than that which we have found. Dulong also determined the refractivity of air, and, allowing for that of the small percentage of carbon dioxide, it is precisely the mean of that of its constituents, taken in the proportion in which they are present.

Returning to the results of Mascart and Lorenz, we have for the D lines:—

	Air.	Nitrogen.	Oxygen.
Mascart	1	1.0178	—
Lorenz	1	—	0.9347

From these data of Mascart and Lorenz it is possible to calculate the refractivity of air:—

$$(1.0178 \times 79.1) + (0.9347 \times 20.9) = 100.15.$$

There is reason to doubt the purity of Lorenz's oxygen. He heated mercuric oxide, of which he does not give the method of preparation; it may have contained oxides of nitrogen; and for some reason, not explained, he passed the gas through a vacuous porcelain tube, presumably red-hot, which, as recent experiments of Messrs. Bone and Jerdan have shown (*Chem. Soc. Trans.*, 1897, p. 42), is not impervious to furnace gases. Dulong, on the other hand, who, as already remarked, prepared his oxygen from chlorate, obtained the number 0.924 for white light, coincident with our determinations.

The refractive index of air, calculated from our determinations, viz.,—

Oxygen	0.9243
Nitrogen	1.0163
Argon	0.9596

and the densities of the constituent gases ("Argon," *Phil. Trans.*, A, 1895, p. 202, footnote), gives the following numbers:—

$$(1.0163 \times 78.15) + (0.9243 \times 20.91) + (0.9596 \times 0.94) = 99.653.$$

Observers sometimes find the percentage of oxygen in air to be about 20.98, or even 21.0. This would hardly affect the result; with 20.96 per cent of oxygen the calculated refractivity is 99.647, instead of 99.653.

There can be no doubt as to the refractivity of oxygen from our ratios, as well as from Dulong's determinations.

* The dispersions for these gases are so small as not to affect the ratios of these numbers (*Compt. Rend.*, 1874, lxxviii., p. 621).

The question is as regards nitrogen. It would require the refractivity of nitrogen to be 1'208, a number greatly above any of our values, in order that the sum of the refractivities of oxygen, nitrogen, and argon should equal 100. The presence of argon would also make an almost inappreciable difference. Taking Mascart's determination of the refractivity of atmospheric nitrogen to be correct, that of pure nitrogen would be 1'0181, instead of 1'0178. And an error in the refractivity of argon would also not affect the result, inasmuch as the total amount of argon is so small.

We are thus driven to conclude that the refractivity of the mixture, air, is somewhat less than that of the sum of the refractivities of its constituents, taken in the proportion in which they occur.

It appeared advisable to try other mixtures; and a mixture of hydrogen and helium was first selected, because these are both very "perfect" gases, inasmuch as their critical points lie very low. It was to be expected that if a difference between calculated and found values should exist, it should be of the inverse character to that of a mixture of oxygen and nitrogen, for they are two somewhat "imperfect" gases. The result has borne out this idea.

A mixture was made of 20'60 c.c. of hydrogen and of 20'12 c.c. of helium free from argon, and of the density 1'960; and with the refractivity of the mixture those of hydrogen and helium were compared. Taking the refractivity of the mixture as unity, the following ratios were found:—

Hydrogen/mixture ..	1'5977	Mean 1'5967
	1'5957	
Helium/mixture ..	0'4513	Mean 0'4495
	0'4478	

The calculated values are—

$$\frac{(0'04495 \times 20'12)}{40'72} = 22'21.$$

$$\frac{(1'5967 \times 20'60)}{40'72} = \frac{80'87}{102'99}.$$

Here the calculated value of the refractivity of the mixture is 3 per cent higher than the found value, while with air the calculated value is 0'35 per cent too low.

A third experiment was made, in which the "artificial air" was a mixture of 19'13 c.c. of carbon dioxide with 19'29 c.c. of oxygen, both gases supposed to be at 0° and 760 m.m. Again, taking the refractivity of the mixture as unity, we found the following ratio:—

Carbon dioxide/mixture ..	1'2450
Oxygen/mixture ..	0'7525

The calculated values are—

$$\frac{(1'2450 \times 19'13)}{38'42} = 61'99.$$

$$\frac{(0'7525 \times 19'29)}{38'42} = \frac{37'78}{99'77}.$$

Here, as with air, the total refractivity found is less than that calculated. It is true the difference is not great, but we are persuaded that it is real, for it considerably exceeds the error of our several determinations.

The case is not bettered if Lorentz and Lorenz's formula be substituted for Gladstone and Dale's. Using their formula, $n^2 - 1/n^2 + 2$, the calculated result is 99'72 per cent of that found for air.

The coefficient of compressibility of hydrogen is too small, while that of other gases, such as oxygen and nitrogen, is too great. The effect of mixing equal volumes of hydrogen and helium, each of which has too large a coefficient of elasticity, is to cause each to occupy twice the volume that they previously occupied, and to halve

approximately the pressure for each. The pressure is therefore lower than it would be for an absolutely ideal gas, for each gas, hydrogen and helium. The sum of these pressures will accordingly be too low, or, transposing, the sum of the volumes will be too great. The opposite argument holds for air.

Now, in considering volumes we deal not merely with the co-volume, *i.e.*, the space occupied by the molecules, but also with the interstitial space inhabited by the molecules. But the refractive power, if Clausius's deduction from the formula of Lorenz and Lorentz is correct, is a function of the dielectric constant, and hence of the co-volumes of the gases. And here the discrepancy is more easily detected than by any determination of density. It must therefore be concluded that gases are not, as postulated by Dalton, indifferent to one another's presence, but that they modify one another's properties in the same manner as do liquids, though to a different extent. This mutual action at high pressures and small volumes modifies even the volume relations, as recently shown by Dr. Kuenen. And it must persist at low pressures and large volumes, though it may not always be possible to make measurements of pressure and volume accurate enough to lead to its detection. The refractivity, however, seems to be a means delicate enough to be used for this purpose.

HYSTERICAL CHEMISTRY.*

By Prof. H. CARRINGTON BOLTON, Ph.D.

THE bizarre chemical theories, extravagant assumptions, and preposterous claims advanced in the writings of a certain small group of men having some knowledge of chemistry and an intense desire for notoriety seem, to a conservative student of contemporaneous science, to be products of disordered intellects; these pseudo-philosophers call themselves "Monists," and designate their peculiar doctrines as "Unitary Chemistry," but, from its emotional character, it may well be styled Hysterical Chemistry.

Their treatises generally have this feature in common; the authors begin with accepted facts and rational theories, leading to legitimate deductions, but at a certain point they allow their imagination to run riot, and thereafter the treatises follow most eccentric lines. The corner stone of their doctrine is the Unity of Matter, an hypothesis to which there is little objection *per se*, but they push it beyond legitimate bounds and graft on it corollaries of a most fantastic character. These authors spend very little time in laboratories, but claim to be working a revolution in chemistry and physics as well as in the sciences of philosophy and psychology by their publications; they complain that "official chemists" will not do justice to their novel theories. "Our views," writes one, "may appear strange to certain materialistic high priests of nineteenth-century science who imagine they know all and claim to explain everything, although they know and explain nothing," and he appeals for righteous judgment to "impartial men of science, lovers of Nature, who divine her infinite power and know how to decipher her language."

Some of these authors boast that their ultimate object is the transmutation of metals, and, together with certain devotees of theosophy, astrology, and occult science, they have formed the "Alchemical Association of France," an organisation whose workings and affiliations I have discussed elsewhere.

Singularly enough these hysterical chemists flourish chiefly in that enlightened nation that has given to science such men as Lavoisier, Gay-Lussac, Dumas, Berthelot, and Moissan. The chief apostles of this new

* Read to the Washington Section of the American Chemical Society, November 12th, 1897.

school are Louis Charles Emile Vial, F. Ch. Barlet, and François Jollivet-Castelot, and their guiding star is August Strindberg, a Swede. Their publications fall within the last twelve years, but they quote two authors whom they regard as pioneers: Louis Lucas, author of "La Nouvelle Chimie" (Paris, 1854) and Théodore Tiffereau, author of "Les Métaux sont des Corps Composés" (Paris, 1855). The latter is, however, purely an alchemist. To the same school belonged Albert Poisson, who was founder of the "Hermetic Society of France," and died in 1894.* All are cordially endorsed by Paul Sédir, "Papua" (Dr. G. Encausse), and other occultists of high rank.

The Monist philosophers do not accept the elementary substances as chemical entities, and claim the unity of matter even though its forms appear diverse to our senses; they do not believe in identity, and are satisfied when analogies lead to a high degree of resemblance. They oppose these positive analogies to the "poetical and metaphysical concepts of official chemists," and as disciples of Aristotle they do not believe in a constructive resemblance of bodies, but only in the acquisition by bodies of given properties under certain conditions. They further claim that "all bodies and all forces have a common origin, and that chemists can derive one from another *at will*, no matter how diverse the phenomena observed, whether rarefaction or condensation, combination or mixture, attraction or repulsion, association or dissociation, addition or substitution." They are not bound by principles of pre-established harmony, of finality, or other vacillating conceptions; they expect the analogies to be clearly demonstrated, and if the accepted nomenclature disappears so much the better (August Strindberg, "Hortus Merlini; lettres sur la Chimie," Edition de l'Hyperchimie, Paris, 1897).

The Monists take for their motto: "Matter is one, everything comes from one, one is all." They claim for this doctrine great antiquity; it prevailed among the philosophers of India, Chaldea, and Egypt, and the alchemists of the Middle Ages represented it symbolically by a serpent biting its tail. The Monists extend the doctrine of evolution to the chemical elements, and assert that the infinitely small particles of primordial ether unite to form chemical atoms, which by self-evolution generate the diverse elementary bodies. Hydrogen, or perhaps helium, is the starting point. According to Tiffereau the evolution of the metals is accomplished by special microbes called spores.

The views entertained by these philosophers are, however, diverse. M. Vial is not properly a Monist; he prefers to refer all matter to two types, one personified by hydrogen and the other by oxygen; according to him, "heat and cold, which are positive and negative elements represented by hydrogen and oxygen, are the universal agents, the cause of all chemical and physical phenomena, distinct and opposite entities." (L. C. Emile Vial, "La Chaleur et le Froid," Paris, 1884). And yet he inconsistently says, "Hydrogen is the divine principle of all things; by self-doubling it creates oxygen, which, being more condensed, and having generating power, becomes its second half." "Light results from the embrace of the primitive hydrogen with oxygen, and their union gives birth to water; hydrogen is the masculine element, oxygen the feminine, and water, which is the first two-fold type of nature, generates nitrogen by self-doubling. Created by water, nitrogen is succeeded by carbon, fluorine, chlorine, bromine, iodine, phosphorus, and finally sulphur."

In reviewing Vial's essay, Jollivet-Castelot remarks, "This masculine hydrogen and feminine oxygen are veritable monstrosities, in spite of the ingenuity of the hypothesis," and he points out that in attempting to con-

struct a cycle of evolution of the chemical elements Vial is often obliged to alter their atomic weights to adjust them to the desired positions, though he brings forward no experimental proofs in support of these arbitrary changes. This complaint of one Monist by another is very amusing, for their disciples never use chemical tests to prove the presence or absence of a given element, but rely on analogies, the full meaning of which will appear later. Moreover the plaintiff in this case is one who gives the widest possible scope to his own amazingly free imagination.

In a recent work ("L'Amour dans l'Univers," Paris, 1896) Emile Vial explains his position in the following language: Unity is a duality of two opposite halves, just as an apple is composed of two half-apples.

Unity is eternally divisible, and it is precisely this infinite divisibility that constitutes its duality." Applying this view to chemical atoms he writes: "A gaseous atom is a unity formed of two antagonistic principles, one material and the other immaterial."

The mental operations of this transcendental philosopher are evidently conducted in four-dimensional space, and suggest the following catechism:—

What is Matter?—Never mind.

What is mind?—No matter.

What is spirit?—It is immaterial.

F. Ch. Barlet, said to be one of the most eminent occultists in France, and one of the founders of the "Université du Hautes Etudes," a school for cultivating esoteric studies, has published an essay entitled "La Chimie Synthétique" (Paris, 1897) which exhibits the peculiar characteristics of hysterical chemistry. Barlet rehearses at length, and with some skill, the growth of chemical theories from the dualism of Berzelius to the Periodic Law of Mendeléeff, and he gives preference to Sir William Crookes's method of representing the elements in order of their atomic weights by a spiral; this graphic construction he improves (?) by changing the spiral into a horizontal plane, which he then divides into four equal parts by two sectors crossing at right angles. The chemical elements falling within these segments he finds correspond to the four elements of ancient philosophy,

Fire	Air
Earth	Water

and on this correspondence he bases all his subsequent ratiocination. It would not be profitable to follow in detail the mental vagaries which finally lead Barlet to present the following scheme.

		(Fire)		
		H		
	HCl, &c.		Metals.	
	Strong acids.		NH ₃ , &c.	
			Strong bases.	
(Air)	Cl	— C —	N	(Earth)
	ClO, &c.		NO ₃ , &c.	
	Weak acids.		Metallic acids.	
		O		
		(Water)		

Following the opinions of P. Leroy (Eudiste, "Essai sur la Synthèse des Forces Physiques," Paris, 1892, 2 Nos.), he imagines the ether of physics immersed in a medium still more subtle called "Eon." A mixture of ether and eon is the protyle of Crookes and the Astral Light of theosophy; in this medium the four elements are formed under the influence of quintessence, which after all is nothing but eon penetrating ether! Barlet

* Albert Poisson's principal works are: (1) "Théories et Symboles de l'Alchimistes" (Paris, 1891). (2) "Cinq Traités d'Alchimie des Plus Grands Philosophes." Traduit du Latin (Paris, 1890); Nicolas Flamet, Paris, 1892.

concludes his extraordinary disquisition with the following lucid summary:—

The principal doctrines of this philosophy are "the periodical, continual evolution of the subtle into the dense, of æon into ether, of quintessence into matter, of the divine Fire attracted by the desire of Astringency, of the Spirit called by Matter, of the Being evoked by Nothingness; the formidable hierarchy of creatures which extends between the two poles of the Infinite, the cosmic life traversing their development as the fulfilment of that ineffable Mystery which manifests itself in the Inexpressible Absolute!"

"I smiled and said it must be so,
In fact I thought so then,
For there was much I did not know
Of the Whatness of the When."

"All that exists lives, all that exists is evolved and transforms itself" is one of the favourite tenets of the Monists, who designate it by the term "Hylozoism." M. Jollivet-Castelot, a member of the "Groupe Indépendant d'Études Esotériques," is the chief exponent of this doctrine, having devoted to it an entire volume besides chapters in other works. ("La Vie et l'Âme de la Matière; Essai de Physiologie Chimique," Paris, 1894. "L'Hylozoisme; L'Alchimie; Les Chimistes Unitaires," Paris, 1896. "Comment On Devient Alchimiste," Paris, 1897.) That which is commonly called inert or dead matter (metals, minerals, elementary bodies, even the physical atoms), according to Hylozoistic Monists, is not dead, but is possessed of life, analogous to plants and animals, but less active and conspicuous. Thus the three kingdoms of nature, animal, vegetable, and mineral, are brought within the influence of evolution. Some of the extremists go so far as to endow the atoms with feeling, instinct, and will (Fouillée, "Revue des Deux Mondes," Oct. 16th, 1886, p. 895), and maintain that the atom plays the same rôle in chemistry as the cell-germ in physiology. One element generates another by polymerisation, scission, agglomeration, substitution, condensation, &c., communicating to it, by *heredity*, certain properties; these properties, however, are combated by the tendency to variation, the sexual selection of the chemical elements, and perhaps by natural selection acting in the chemical world as in organised beings." The author cited (Jollivet-Castelot) writes of the individuality of mineral beings as proved by their choice of colours, and of their "souls," which, however, are inferior to the souls of plants and animals. "Admitting a human spiritism, there must exist a vegetable and a mineral spiritism, for we know that the entire universe is animated by an evolutive Intelligence organising Matter and condensing Energy." "I will not go so far," writes Jollivet-Castelot, "as to affirm that electricity is the soul. . . . but I venture to say that electricity excites the spiritualistic phenomena, accompanies them, and serves perhaps as an intermediary between conscience and organised matter." Applying these extraordinary views to common physical and chemical phenomena, the Hylozoist states that "Water of crystallisation constitutes a kind of mineral respiration, demonstrating in a special way the Life of Matter. The substance has a positive need of water, absorbs it, utilises it, expels it, and submits to an evident motion of inhalation and exhalation."

This bizarre doctrine is opposed by its disciples to the materialistic theories of scientists who would reduce mental activity to automatic mechanism, and they cite approvingly the profound epigram of Aristotle:

"All motion is a kind of appetite,"

as well as the saying of Leibnitz:

"Nothing in nature is dead."

Great antiquity is claimed for this philosophical system. It served as a foundation for ancient Oriental religions; the priests of Brahma and of Buddha, the hierophants of Chaldea and of the sacred temples of the Egyptian goddess Isis, all maintained that the universe is living,

animated, and evolved. Moreover the alchemists of the Middle Ages and eminent physiologists of ancient and modern times, have uniformly supported these opinions. In a few years the scientific world will doubtless be entirely converted to Hylozoism.

A certain number of these hysterical chemists are devoted to theosophy, and apply its principles to the more practical science; they claim that the true adept has no need of retorts, furnaces, or chemical menstrua, but he is capable of accomplishing the union and separation of atoms by the force of his will and the power of his magical faculties.

(To be continued).

LABORATORY NOTES.

ASBESTOS—COMBUSTION FURNACES.

By H. JERVIS.

It is a trite observation that a body is valuable as it possesses hardness, weight, stability, or other properties desirable for a set purpose. Asbestos possesses properties so varied, and so subject to designed modification, that its limited use in the laboratory may well excite remark.

The most useful property is its comparative stability at high temperatures. This property is all the more valuable because, before heating, the asbestos may be moistened with water and worked into any desirable shape as easily as a piece of clay. Such models when dried, but not ignited, may be roughly handled without detriment. Other of its properties are indifference to sudden heating or cooling, lightness, and toughness. Two other properties, which have frequently to be considered negative ones, are the tendency to laminate when subjected to pressure and the rottenness after strong ignition. It does not seem unreasonable, however, to hope that these properties may be modified.

To illustrate the value of this material we will mention a few of the forms in which it has served us in the laboratory, some of its common uses, and then some new uses, along with a few forms of apparatus its use makes possible.

As fibre, asbestos is used everywhere for filtering liquids, plugging furnace tubes, &c. As woven cloth, it is useful for wrapping round the necks of hot water wash-bottles, though in this respect it is inferior to sheet cork, except that it lasts much longer; the threads, which are easily detached from the piece, are used as string, where hemp or even wire are not permissible; this form is very strong, and of course does not laminate. Millboard, useful kinds of which may vary from the thickness of stout paper to half an inch or more, is used for laying over muffle bottoms, coating porcelain boats so that they may not stick to more fusible surfaces, wrapping round combustion tubes after cutting (if needful) an opening through which to watch the operation.

Two other uses are so intimately connected with the modifications to be described, that they may be mentioned separately.

White (CHEMICAL NEWS, xlv., 65) describes an asbestos stopper made by separating the asbestos into fine threads, moistening with water, twisting into the cylinder of a steel mortar, and pressing in a vice. Here the tendency to laminate is much lessened by using the long threads, and the stoppers are serviceable for a few times, but they are too fragile to bear the pull and push of constant use. As an adjunct to indiarubber, we have used a thick washer of asbestos millboard fitted to the smaller end of the stopper, and kept in position by turning the ends of the glass tube outwards. The millboard contains oily matter, which may be removed by simply igniting in a muffle. The washers are much less subject to lamination if they be

soaked in some decomposable metallic salt before ignition. A mixture of chromic and sulphuric acid gives good results, but the washers must be only just soaked, not digested.

Stoke (CHEMICAL NEWS, lvii., 150) suggested that furnace tiles, if cut from asbestos millboard, would form a lighter, hotter, and less breakable covering for a combustion-tube than those made of fire-clay. The suggestion has much merit. In practice, however, the teeth of the tiles made "rotten" by heat are readily damaged during the unavoidable putting on and off which oftentimes attends combustions.

It may be taken as a general rule that, when intended for exposure to heat and handling, an asbestos model should be made as free as possible from edges and corners. In keeping with this dogma we recommend the shape shown in Fig. 1. Its position on the furnace is

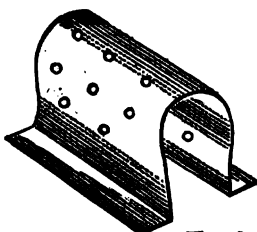


FIG 1

sufficiently obvious. It is necessary only to describe its construction.

A piece of millboard (8" x 4" x 10") is perforated with glycerined cork-borers, as shown. It is then nicked about an inch from each short end, and laid in water until quite saturated. Bend from the nicks through 90°, and then lay the centre of the asbestos over a bottle (2" diam.), and bend it to the bottle's shape. Very little force is necessary and very little should be used, or the holes will be closed somewhat. Remove the bottle, press the ledges of the model on to the table, and amend irregularities of shape. The drying may best be done by removing the cover to its furnace, setting it in position on the supports, and pressing it well thereto. When quite dried by the lighted furnace it will be found to have retained its shape and to need a knife to separate it from the iron supports.

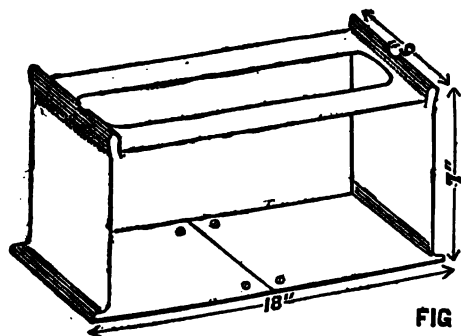


FIG 2

Our experience has been that the temperature is greater with these covers than with fire-clay tiles. When the temperature needs to be unusually high, a second larger cover, but similar in other respects, is placed over the first, and can be moved along the tube at will.

In appearance and weight the new cover is a great improvement. To satisfy curiosity it was found that the twenty-four tiles previously used, weighing 13 lbs., had been replaced by three covers weighing 1 lb. This of itself is merely a detail when the ordinary form of furnace

is satisfactorily at work, but it opens the way to the general adoption of a lighter form of furnace.

Combustion furnaces of thin sheet iron have been previously described. We submit the form shown in Fig. 2. It is represented screwed to the table. It was made from a single sheet of mild steel 5 feet long and about one thirty-second of an inch thick, during a meal-time (1½ hours) in the laboratory. The outer pieces are not cut away, but nicked merely and bent under. The centre-piece was cut out with a sharp chisel, and the tube nests made with a file. The bending was done chiefly over table-edges and cupboard-doors. When used with the afore-mentioned covers it forms an apparatus both cheap and effective.

A common support for combustion-tubes used temporarily is a Y-shaped stiff wire, sliding in a wooden base. Such an arrangement may be very readily converted into an efficient combustion-furnace, having the appearance shown in Fig. 3.

The arrangement supporting the tube and cover is made from a piece of steel, circular in section and tapering to the ends, to which are attached the wires. Such pieces with threaded holes are returned to steel-works in large quantities along with "spindle scrap"; I believe they are spindles.

As we have become so mixed up with combustion-furnaces, it may be as well to use its accessories to illustrate some further uses of asbestos.

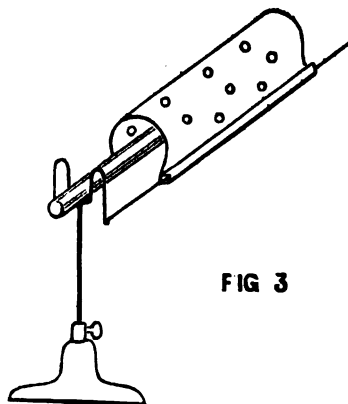


FIG 3

Tubes of asbestos are out of the question at present. The Y-shaped movable tap for Bunsen burners, giving a long flame, may very easily be made from asbestos, and, as they need only be dried, they stand hard wear for a long time. After soaking the tips in suitable solutions they show flame colourations very well. Two pieces of thin millboard are cut, wetted, and shaped; a thin slip of glycerined wood or glass placed between the upper end preserves an even opening, and may be readily removed when the rest is dry. To fit it on to the burner it is necessary only to wet the circular end, place it in position, and press it.

The furnace cover serves very well, when suspended by one edge, for augmenting the heat of a blowpipe flame when working hard combustion-tubing. It is possible thus to make bulbs and bottles from material which with the unaided flame could be drawn out only with difficulty after prolonged heating.

There are other uses of asbestos peculiar to personal occupations, and of no general interest. One need not grow hyperbolic in its praise. When its obedience to one's fingers is once understood it becomes more interesting than a kindergarten lesson to shape the material to its manifold uses.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, December 16th, 1897.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. James C. Philip, Edward Rosling, and Frank T. Addyman were formally admitted Fellows of the Society.

The PRESIDENT announced that the following had been recommended by Council for election as Foreign Members, to be balloted for at the next meeting, January 20th, 1898.

Prof. Remsen, Baltimore, U.S.A.; Prof. Troost, Paris; Prof. Moissan, Paris; Prof. Raoult, Grenoble; Prof. Ostwald, Leipzig; Prof. Curtius, Bonn; Prof. Mensutkin, St. Petersburg; Prof. Markownikow, St. Petersburg; Prof. Arrhenius, Stockholm; Prof. Waage, Christiania; Prof. Franchimont, Leyden; Prof. van der Waals, Amsterdam; Prof. Spring, Liège; Prof. Körner, Milan.

Certificates were read for the first time in favour of Messrs. E. L. Allhusen, B.Sc., Geological Survey, Perth, W.A.; B. S. Bull, M.A., B.Sc., Ph.D., 49, Devonshire Road, Greenwich; T. H. Hille, 6, Elliot Park, Blackheath; J. E. Miller, Holmoor, Patrington, Hull; G. T. Morgan, 35A, Russell Road, Kensington, W.; W. E. Moss, B.A., Burnthwaite, Bolton; H. Poole, 323, W. 34th Street, New York; W. Richards, Old Elvet, Durham.

The following papers were read:—

*126. "Stereochemistry of Unsaturated Compounds. Part I. Esterification of Substituted Acrylic Acids." By JOHN J. SUDBOROUGH and LORENZO L. LLOYD.

In order to determine whether there is any general rule applying to the esterification of unsaturated acids, similar to those discussed by Mensutkin for fatty acids, and by Victor Meyer and Sudborough for aromatic acids, the authors have investigated the following acids: cinnamic acid, allocinnamic acid, atropic acid, ortho-, meta-, and para-nitrocinnamic acids, α -bromocinnamic acid, α -bromallocinnamic acid, two β -bromocinnamic acids, two α - β -dibromocinnamic acids, α - β -dichlorocinnamic acid, α - β -di-iodocinnamic acid, α -cyanocinnamic acid, α -cyano-meta- and α -cyano-ortho-nitrocinnamic acid, α -phenylcinnamic acid, α -phenylallocinnamic acid, six α -phenyl-nitrocinnamic acids, triphenylacrylic acid, α - β -di-iodo-acrylic acid. Half a gram. of each acid was boiled for an hour with 10 c.c. of a 3 per cent. solution of hydrogen chloride in methylic alcohol, and the amount of ester formed determined in the usual manner.

The authors draw the following conclusions from the results which they have obtained:—

(1) Unsaturated acids of the types,



yield but small amounts of ester when treated in the manner described.

This furnishes a ready method for distinguishing stereoisomeric acids.

(1) $H(Y)C:C(X)CO_2H$ and (2) $H(Y)C:C(CO_2H)X$.

As acids of the trans-type (1) are esterified with difficulty, and acids of the cis-type (2) with ease by the above method, it is probable that the method can also be used for separating mixtures of such acids, in very much the same manner that diortho-substituted benzoic acids can be separated from their isomers.

(2) An α -substituted acrylic acid, $CO_2HC(X):CH_2$, is more difficult to esterify than a β -substituted acid, $CO_2HC(H):CHX$.

(3) In certain substituted cinnamic acids, a nitro-group in the ortho-position appears to have a retarding influence. The results obtained are completely in accord

with those published by Anschütz (*Ber.*, 1897, xxx., 2652) whilst the present work was still being carried out.

The constitution of camphoric acid is discussed in the light of the results contained in the paper.

The authors think it desirable to carry out similar researches with substituted acids; they have already found that dibromosuccinic acid, phenyldibromopropionic acid and its nitro-derivatives, yield but little ester when boiled for an hour with a 3 per cent. solution of hydrogen chloride in methylic alcohol. Several new methylic esters have been obtained, and are described in the paper.

*127. "Formation and Hydrolysis of Esters." By JOHN J. SUDBOROUGH, Ph.D., D.Sc., and MARTIN E. FRIELMANN, B.Sc.

From the researches of Mensutkin (*Annalen*, 1879, cxcv., 334, 1879, cxcvii., 193) on the esterification of fatty acids, of Victor Meyer and Sudborough (*Ber.*, 1894, xxvii., 510, 1890, 3146) on substituted benzoic acids, and of Sudborough and Lloyd (preceding abstract) and Anschütz (*Ber.*, 1897, xxx., 2652) on unsaturated acids, the authors consider it proved beyond doubt that stereochemical influences play a most important part in the esterification of an acid by means of an alcohol and hydrogen chloride. Researches by Kellas (*Z. Physik. Chem.*, 1897, xxv., 221) on the esterification of monosubstituted benzoic acids, and of Sell (*Trans.*, 1897, lxxi., 1070) on substituted pyridinecarboxylic acids also support the same conclusion. In the benzoic and acrylic acid series, the chemical nature of the substituting groups has but little, if any, influence on the retardation of esterification. CH_3 , F , Cl , Br , I , NO_2 , OH and CO_2H groups all act in the same manner; the radicle weight or volume, however, affects the retardation to a certain extent (Meyer, *Ber.*, 1895, xxviii., 1259, and Kellas, *loc. cit.*).

One would therefore conclude that in the fatty series, too, the chemical nature of the substituting groups would have but little, if any, apparent influence. A tertiary fatty acid, however, such as trimethylacetic acid, yields but little ester when heated with alcohol (Mensutkin), whilst the similarly constituted trichloroacetic acid is most readily esterified, in fact, much more readily than acetic, mono- or di-chloroacetic acids (Lichty, *Am. Chem. Journ.*, 1896, xviii., 590). This difference in behaviour we can only attribute to the enormous increase in strength of the acid.

	K.		K.
(1) Acetic acid	0.0018	(3) Dichloroacetic acid	5.14
(2) Chloroacetic acid	0.155	(4) Trichloroacetic acid	121.0

Lichty's researches show that these four acids follow exactly the order given above as regards the ease with which they are esterified. The amounts of ester formed at the end of any given time by no means bear the same ratio to one another as do the affinity constants of the corresponding acids.

	After 1 min.	1 hour.	2 hours.
Chloroacetic	1.78	41.89	57.33 per cent
Dichloroacetic	4.56	56.49	62.34 "
Trichloroacetic	9.99	59.39	66.18 "

It would appear then that some other factor is introduced which tends to hinder the formation of the esters, and this factor the authors consider to be the stereochemical influence of the chlorine atoms. The general conclusion drawn is that in the conversion of an acid into its ester by the action of an alcohol, either with or without hydrogen chloride, the rate of esterification is determined by two factors:—1. The configuration of the acid; in other words, the presence of substituting groups situated close to the carboxylic group, which always tend to hinder esterification. 2. The strength of the acid as determined by its affinity constant. In most of the cases which have been more closely studied, the first factor is the more prominent, and obscures to a large extent, the

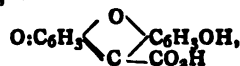
influence of the second factor. In exceptional cases, however, namely, in acids the strength of which has been enormously increased, the second factor becomes the more prominent, and the influence of configuration is cloaked. In all cases, however, we must suppose the two factors to be in force, and it is only in acids which have the same affinity constant that the true effect of stereochemical influence can be looked for.

No general systematic study of the rate of hydrolysis of esters appears to have been attempted. Victor Meyer (*Ber.*, 1895, xxviii., 1263) formulated the generalisation that those esters which are the most difficult to form are also the most difficult to hydrolyse. This view was also held by Wegscheider (*Ber.*, 1895, xxviii., 2536) and Brühl, but according to the more recent investigations of Kellas, this generalisation is not strictly correct. Kellas (*loc. cit.*) stated the rate of hydrolysis of the methyl salts of mono-substituted benzoic acids by means of alkali. He found that in any given series the ortho-compound is always the most difficult to hydrolyse, the para- occupies an intermediate position, and the meta- is most readily hydrolysed (with the exception of the nitro-compounds). It is stated, however, that methylic orthonitrobenzoate is much more readily hydrolysed than methylic orthotoluate, although the nitro-ester is formed much more slowly than the toluate. This and similar results noted by Kellas, for example, the fact that methylic benzoate is difficult to hydrolyse, can be accounted for by the introduction of the affinity of the acid as a factor. For orthonitrobenzoic acid $K=0.016$, for orthotoluic acid $K=0.012$, and for benzoic acid $K=0.006$. The results obtained by Hjelt (*Ber.*, 1896, xxix., 1864) on the hydrolysis of the ethylic salt of substituted malonic acids also lead to the same conclusions. They indicate that here, also, the rate of hydrolysis does not depend merely on the stereochemistry of the ester molecule. If the affinity constants are taken into consideration, the obstruction due to the substituting groups is much more pronounced (with the exception of ethylic allylmalonate). Investigations by the authors on the hydrolysis of the ethylic salts of acetic, methyl-, dimethyl-, and trimethylacetic acids by the aid of sodium hydroxide, prove that here, where the affinity constants of the acids vary but little—acetic acid $K=0.0018$; propionic acid $K=0.00134$; isobutyric acid $K=0.00144$, and trimethylacetic acid (unknown, but at any rate very small, since the salts of this acid are extremely unstable)—the rates of hydrolysis are what would be expected from the introduction of the substituting groups—

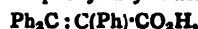
consider that these generalisations can be proved or refuted only by a much more careful study of the hydrolysis of numerous series of esters. They themselves intend investigating the ethylic salts of substituted (alkylated) succinic acids, as the strengths of these acids differ but little, and therefore the results obtained should throw light upon the retarding effect of different alkyl groups.

DISCUSSION.

Dr. HEWITT said that the fact that hydroxydifluorocarboxylic acid,—



yields scarcely any ethyl ester, even after prolonged boiling with 9 parts by weight of ethyl alcohol and 1 part of concentrated sulphuric acid, is easily explicable in the light of Dr. Sudborough's work. Hydroxydifluorocarboxylic acid is a tri-substituted acrylic acid, and hence should not easily esterify. On the other hand, Victor Meyer showed that triphenylacrylic acid,—



yielded an ester by prolonged boiling with methyl alcohol and a stream of hydrogen chloride. Under similar circumstances, triphenyl acetic acid, $(\text{C}_6\text{H}_5)_3\text{CO}_2\text{H}$, furnished very little ester.

Dr. SHIELDS called attention to the numbers, quoted by the authors of the papers, representing the percentage amount of etherification after one minute, one hour, and two hours, and remarked that, whereas all the limiting values were approximately the same after sufficient time had elapsed, a considerable difference existed between the numbers when the amount of transformation was measured after the lapse of only one minute. If it had been possible to obtain the corresponding values after one second, he thought the difference would have been still greater, and that in any serious attempt to study parallelism between the velocity of etherification and the affinity constants of the acids, it would be necessary to determine the velocity constant of the reaction or the initial rate of etherification.

*128. "A New Method of Determining Freezing-points in very Dilute Solution." By MEYER WILDERMAN, Ph.D. The chief condition for obtaining correct determinations of the freezing-point is the establishment of equilibrium between the solid and the liquid part of the

	Hours.	0.25	0.5	1	1.5	2.5	3	4	4½	8	10	22.5	70	
Ethylic acetate	19.6	28	42	—	—	66	—	—	—	82.6	85.4	—	—	per cent.
" propionate	14.4	19.6	—	35.6	—	50.8	—	—	61.2	—	—	—	—	" "
" isobutyrate	5.2	7.2	11.4	—	19.2	—	—	—	—	—	—	61	—	" "
" trimethylacetate ..	—	—	—	2.4	—	—	6.8	—	—	8.8	—	—	19.6	" "

With the esters of chlorinated and brominated acetic acids, the reverse is true, and all are decomposed much more readily than ethylic acetate itself. Comparative experiments with these esters are being undertaken by the authors.

The general conclusion arrived at is, therefore, that in the hydrolysis of esters by the aid of an alkali the same two factors operate as in esterification. The strength of the acid which is formed by the hydrolysis of the ester seems, however, to play a more important part than it did in esterification, and this accounts for the fact that it does not always follow that esters which are most readily formed are the most readily hydrolysed.

Other factors are apparently introduced when esters are hydrolysed by the aid of hydrochloric acid, as it has been proved that esters by no means arrange themselves in the same order when hydrolysed by an alkali as when hydrolysed by an acid (Hemphill, *Z. physik. Chem.*, 1894, xiii., 561; Löwenhertz, *ibid.*, 1894, vi., 389; Van Dyken, *Rec. Trav. Chim.*, 1895, xiv., 106). The authors

heterogeneous system. Starting from the properties of "perfect" equilibrium, and from the equations for velocity of ice-melting, ice-separation, and Newton's equation for cooling, all the conditions necessary for a successful experiment, previously laid down empirically by the late P. B. Lewis and then by the author, are now deduced from theoretical considerations. It is shown how to arrange the equilibrium with an accuracy of $0.000002-0.00006^\circ$, and even greater. The freezing-point method, which has been hitherto a conglomeration of empirical rules, is thus placed on a physico-mathematical basis, and the experimental error of all previous methods can be calculated. The second important point in a freezing-point method is a correct and very detailed knowledge of the registering instrument. A very careful study of the errors of mercury thermometers has been made for a long time, and an account of this is given. This paper is a continuation of that by P. B. Lewis (*Trans.*, 1895, lxvii., 1).

DISCUSSION.

Mr. PICKERING expressed his disappointment that the

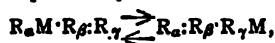
author had not made any statement as to what his "new method" was. All that he had given was a summary, in the form of an equation, of the various inaccuracies inherent in freezing-point determinations generally. These inaccuracies were well known, and it was of great importance that they should be reduced to a minimum; but it was impossible to say whether the author's new method succeeded in doing this or not until a description of the method was given.

Dr. SHIELDS remarked that the literature and controversies on the vexed question of the accurate determination of the freezing-point of dilute solutions would already fill a large volume. Since the subject was one of great importance in connection with the modern theory of solutions, any new contribution which was likely to lead to a final settlement of the conflicting views was welcome. He regretted that want of time had prevented Dr. Wilderman from giving a fuller account of his paper, but the method of attack adopted by the author was undoubtedly a step in the right direction, and he thought that the study of the time reaction and of equilibrium in freezing solutions would lead, if it had not already in Dr. Wilderman's hands led, to important advances.

Dr. WILDERMAN, in reply, stated that methods were given in the paper for obtaining the separated ice in fine films throughout the liquid. In answer to Mr. Pickering, he said that the value of the equations he laid down was, that by them the errors due to equilibrium in the methods of other investigators could be estimated, and their results re-calculated. As an illustration, one of the most accurate of the recent methods was re-calculated, and its error found to be 160 times greater than his own. He had presented, for the first time, the physico-mathematical theory of freezing-point determinations in such a form that future experimenters could arrange with ease the equilibrium to any degree of accuracy required.

*129. "A possible Basis of Generalisation of Isomeric Changes in Organic Compounds." By ARTHUR LAFWORTH, D.Sc.

In this paper the author points out that many isomeric changes, hitherto regarded as belonging to different types, may be formulated as special cases of a general form, which may be expressed by the reversible equation—



representing a labile group moving from an α -atom to a γ -atom, the necessary re-arrangement of single and double bindings taking place between the three atoms R_a , R_β , and R_γ . A conventional "mechanical" representation of the change is given for the special case where R_a , R_β , and R_γ are carbon atoms.

Examples, for the most part derived from "tautomeric" and "desmotropic" substances, such as acetoacetic ether, cyanic acid, nitroso-compounds, &c., are shown to be of the above type.

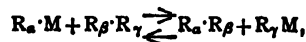
Extensions of the above special form are deduced, and the general conclusion is arrived at, that in a chain of alternately singly- and doubly-linked atoms, either (1) a labile group may become successively attached to alternate atoms, or (2) an exchange of labile groups in γ - (i.e., meta-) positions may occur. These deductions are shown to be confirmed by the behaviour of benzenoid compounds, &c., the fully justifiable assumption being made that benzenoid compounds may act as if possessing Kekulé's formula. Special reference is made to nitrophenol, ortho-hydroxyazobenzene, the sulphonic derivatives of aniline and of β -naphthol, and also to the changes of methyl-aniline into paratoluidine, and of hydrazobenzene into benzidine, which are all shown to exhibit changes in complete accordance with the author's views.

Further modification of the general formula for the case where R_β and R_γ are singly, instead of doubly, linked, is shown to lead to the form—



which represents on the one hand the great majority of simple molecular decompositions of organic compounds, and on the other the formation of the ordinary addition products from unsaturated substances. The production of substitution derivatives, &c., by successive addition and isomeric change (compare Armstrong, *Trans.*, 1887, li., 258), as well as some simple cases of hydrolysis, are then discussed.

From the last formula the following type is shown to be at once derivable:—



which represents the ordinary process of substitution in saturated compounds (compare Armstrong, *loc. cit.*, and Williamson, "Theory of Etherification, 1851).

With the help of the foregoing principles, the author shows that the production of meta-di-derivatives from certain mono-derivatives of benzene may be consistently explained on the basis of Armstrong's suggestion (*loc. cit.*) that in such mono-derivatives addition of the acting agent to the side group precedes substitution. Further, this assumption at once explains the replacement (by a substituting group) of side groups, which otherwise afford meta-derivatives.

Some apparent exceptions to the foregoing principle are next dealt with, and it is shown that little difficulty exists in relegating to the above type the production of, for example, pinacolines from pinacones, acetanilide from acetophenone-oxine, benzoic acid from benzil, &c., on suppositions which, for the most part, the author has not been the first to make, and which mostly involve the intermediate production of ring-compounds.

It is further shown that the following type of change is possible, but by no means necessarily, independent of the above, viz.,



(where R_β represents an atom possessing a "residual

affinity" of two units), and a possible method of harmonising this with the other type is discussed in detail.

Finally, it is pointed out that, although a brief dissociation between the labile group and its attached atom must be assumed in order to account for the above changes, cases involving electrolytic dissociation or partial destruction of the labile substance necessarily involve probable changes other than an $\alpha\gamma$ -isomeric change, and should therefore be as far as possible avoided in investigating the applicability of the foregoing generalisation.

DISCUSSION.

Dr. WYNNNE regretted that the author, in reading his paper, had thought it more important to enumerate the various well-known cases of tautomerism, than to give the explanation he had devised to account for this property. So far as could be gathered the author sought to connect those reactions in which a single radicle is transferred from one to the other of positions relatively 1:3 accompanied by a change of structure in the chain, with those in which mutual exchange of radicles in positions relatively 1:3 is brought about without any structural rearrangement. It was difficult to see wherein the analogy lay, but even if the author's contention were adopted, it was open to question whether anything had been thereby gained. The usual statement that ortho-, or para-, or both di-derivatives result from compounds of the phenylsulphamic acid type expressed the facts at least as clearly as the author's emendation, wherein the reaction is represented as being brought about by the radicle moving from any given position to the next but one. What was to be desired was an explanation of the cause of the mutual transference, not a re-statement of the fact of its occurrence.

The author's view of the formation of metanitrobenzene-sulphonic acid surely needed more explanation than the reaction for which it was put forward to account, and the same might be said of the arguments brought forward to explain the production of metasulphonic acids from aniline derivatives. A striking case of the production of such acids is exhibited by dimethylaniline, which with ordinary sulphuric acid gives the para-, whilst with fuming sulphuric acid, a mixture of the meta- and para-sulphonic acids is obtained. Yet, according to the author's views, the meta-sulphonic acid should be the sole product in either case, since he considers that compounds of the phenylsulphamic acid type—which dimethylaniline can form—give rise to metasulphonic acids.

Again the order in which isomeric sulphonic acids seem to be formed in the case of β -naphthol had been adduced by the author in support of his views, but it was not difficult to quote cases in which it appeared to be inapplicable. Erdmann had shown that 1:4-, 1:4', and 1:3'- α -naphthylaminesulphonic acids are the products of the sulphonation of α -naphthylamine, and it was not easy to see how the production of the last-named and most stable of these acids could be accounted for by the author, even if his views did not beg the question of the mode in which isomeric sulphonic acids are formed, as reference to Erdmann's paper would show.

He was, moreover, unable to follow the author in tracing an analogy between the changes involving the production of ethylene from ethyl bromide, and of ethyl bromide from ethylene on the one hand, and those occurring in tautomeric compounds on the other; an analogy, that is, between reactions involving scission of the chain, and those in which none occurs. Markownikow's rule embraced what is known of such reactions, and the author's re-statement of some of the facts did not seem to add to, or explain, that well-known summary.

Professor COLLIE said that this generalisation of Dr. Lapworth's appeared to him to be one of great importance. Any simple rule that would explain not only all cases of isomeric change amongst paraffinoid as well as amongst benzenoid derivatives, but also the hydrolysis of esters, the decomposition of diazo-compounds, the formation of meta-derivatives, &c., &c., was one which certainly claimed the attention of chemists. Moreover, it received much support from a stereochemical point of view; and if Dr. Lapworth's first equation be allowed, then the rest of the argument was a clearly worked out, logical deduction.

Dr. KIPPING agreed with the last speaker, that the paper under discussion was one of great interest and importance. Starting from a simple case of tautomeric change, by an ingenious and logical application of views already widely accepted, Dr. Lapworth had attempted to account for, and to bring into line, a very large number of cases of isomeric change, which before seemed to be complex, obscure, and not related to one another in any way. If this generalisation were possible, as it certainly seemed to be from the formulæ and explanations which Dr. Lapworth had advanced, his paper deserved, and would no doubt receive, the careful consideration of chemists.

Mr. A. G. BLOXAM asked whether, in the event of such tautomerism as that referred to by Dr. Lapworth occurring in di-substitution products, the second group would exert any preventive action on the wandering of the first group. Thus, supposing that a sulphonic group tended to wander into the next following γ -position, which, however, was already occupied by another group, would such a state of affairs determine the non-existence of a tautomeric form?

Dr. LAPWORTH, in replying, regretted that the time at his disposal had prevented him from saying all he had wished. He had tried, in the first instance, to show that a great many isomeric changes, including those of elimination of two groups attached to contiguous carbon atoms and also of most changes in benzenoid compounds,

might be written in a form derived from that representing the relationship between the two forms of simple tautomeric substances, and had shown in the paper that they might be represented on exactly similar stereo-chemical bases. The latter he had omitted to give in reading, as it involved the introduction of the usual hypothetical space-relationships of carbon atoms and the idea of dissociation as distinct from decomposition, whilst the paper intended to call attention to what appeared to be a general type of change, logically deducible from a well-known form.

With regard to the production of benzenoid substitution derivatives, he had already pointed out that, as a rule, where substitution in the benzene nucleus undoubtedly occurs, ortho- and para-derivatives are the chief products, and that this fact, as also the question of the stability of a particular benzene derivative, must depend on some principle not intimately connected with the mechanism of isomeric change. A change, analogous to that of compounds of the aniline hydrogen sulphate type would afford meta-derivatives, and evidence of this is afforded by the production of metanitrilaniline when aniline nitrate is treated with cold sulphuric acid; the production of ortho- and para-derivatives may then be due either to direct substitution in the ring, or to isomeric change of compounds of the phenylsulphamic acid or methylaniline type. Hence the formation of any particular acid from aniline, dimethylaniline, α - and β -naphthylamines, &c., is not difficult to account for, although the exact details of the production, or any one of them, would require a special discussion.

The convenience of employing the above formulæ and the apparently very general applicability of the type of change to which the author desired to draw attention, would only be understood when it was applied to cases which, as the author shows in his paper, are difficult to discuss on the usual bases.

In reply to Mr. Bloxam, the author said that the paper dealt with cases in which the γ -position was already occupied, and in the special case alluded to, the author's view made it clear that no entirely preventive action would be exercised, and this would appear to be in accordance with known cases.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 21, November 22, 1897.

Influence of Temperature on the Rotatory Power of Liquids.—Ph. A. Guye and Mlle. E. Aston. — The authors find that there are a few exceptions to the general rule that the specific rotatory power of a liquid diminishes progressively, with an increase of temperature, without any sudden variation when the liquid passes to the gaseous state. The most interesting case up to the present appears to be amyllic alcohol, in which the rotatory power at first diminishes as the temperature rises, only to increase again as the boiling-point is reached.

On Chlorocyanamide, $C_2N_3(NH_2)_2Cl$.—Paul Le-mout. — When cyanuric chloride is digested in the cold with an ammoniacal solution the latter disappears completely,—a salt of ammonia is formed and a white powder deposited, very slightly soluble in water: this is monochlorised cyanuramide, the chlorocyanamide of Liebig. This impure product is dissolved in a large quantity of boiling water; on cooling, the filtered solution deposits fine needle-like crystals of the pure substance. Its molecular heat of combustion, at a constant volume, is 401.3

cal., and at a constant pressure 400·3 cal. The molecular heat of formation at constant pressure is therefore 25·6 cal.

Contribution to the Study of the Nitrification of Soils.—Th. Schloësing, jun.—This paper will be inserted in full.

No. 22, November 29, 1897.

On a New Method of Preparing Carbides by the Action of Carbide of Calcium on the Oxides.—Henri Moissan.—Besides its curious action on water, carbide of calcium acts as a powerful reducing agent, and, thanks to this property, can furnish a number of new compounds by double reaction, but only when in contact with the body in a liquid state, or a state of fusion at a sufficiently high temperature. In a state of fusion it acts energetically on the oxides, and if the metal will not unite with carbon—such as lead, tin, and bismuth—it is set at liberty, and can be separated or combined with other bodies, according to the condition of the experiment. If the metal or metalloid is capable of being carburised a double decomposition takes place, according to the formula—



in which R represents the metal and x a variable number of atoms of carbon. By this method definite crystallised carbides of aluminium, manganese, chromium, molybdenum, silicon, &c., have been prepared.

Nomination.—M. Ditte was elected a member of the Chemical Section in succession to the late M. Schützenberger.

Influence of Altitude and Warmth on the Decomposition of Oxalic Acid by Sunlight.—M. and Mme. Vallot.—In a series of experiments, made simultaneously at Chamounix (altitude 1995 metres) and at Montanvert (altitude 1925 metres), considerably more decomposition took place at the higher station, the mean difference being 2·1 to 1 for a difference of level of 830 metres. Glass stops a large proportion of the chemical rays. The result of the experiments shows that temperature plays a more considerable part than would have been at first believed, but in the combined action of the two the greatest influence is exercised by light.

On the Alcoholic Isocyanurates, and the Constitutional Formula of Cyanuric Acid.—Paul Lemoult.—Not suitable for abstraction.

On Quinones and Hydroquinones.—Amand Valeur.—The author has studied the quinones and hydroquinones from the thermo-chemical point of view, and finds that the difference of the heats of formation of toluquinone and ordinary quinone is considerable (14·8 cal.), while that between benzene and toluene is only 6·4 cal. By neglecting the first terms of the two series, it is noticed that the homologous relations are parallel between the phenols and the quinones.

On the Absorption of Organic Matter by Roots.—Jules Laurent.—Grains of maize were sterilised by being kept in a weak solution of bichloride of mercury for two hours, and were then cultivated in a solution of, distilled water 1 litre, nitrate of lime 1 grm., chloride of potassium 0·25 grm., sulphate of magnesia 0·25 grm., mono-potassic phosphate 0·25 grm., and a few drops of a dilute solution of perchloride of iron: the maize developed normally, even to the complete opening of the flowers. On adding to the solution a known weight of glucose, and again of inverted sugar, it is found that the quantity of sugar absorbed is in *rapport* with the dry weight of the plant.

On the Analysis of Silicates. — A. Leclère. — This paper will be inserted in full.

Journal de Pharmacie et de Chimie,
Series 6, vol vi., No. 9.

Parisian Hospital Bread.—M. Balland.—Not suitable for abstraction.

On Retamine.—J. Battandier and Th. Malosse.—The molecular weight of retamine, determined by tonometry after being raised to boiling-point in solution in ethylic alcohol, has been found to be 269·4 and 268·3. The estimation of the carbon, hydrogen, and nitrogen has given C = 72·08, H = 10·69, N = 11·1, which corresponds almost to $C_{15}H_{26}N_2O$. Two bromhydrates have been prepared corresponding to $C_{15}H_{26}N_2O \cdot HBr$ and $C_{15}H_{26}N_2O \cdot 2HBr$. Beautiful crystals of iodhydrate have been prepared, which appear to correspond to the formula $C_{15}H_{26}N_2O \cdot 2HI$. Sulphates corresponding to $C_{15}H_{26}N_2O \cdot H_2SO_4 \cdot (H_2O)_x$, where $x=5$ for salts crystallised from aqueous solution, have been produced. Retamine combined with one equivalent of acid no longer colours phenolphthalein. The salts with two equivalents of acid, treated in aqueous solution with one equivalent of caustic alkali, are transformed into salts with one equivalent of acid, $R_2HBr + NaOH = R \cdot HBr + NaBr + H_2O$. In this transformation, one molecule of soda corresponds with one molecule of retamine; the end of the reaction is indicated by phenolphthalein. Retamine will give neutral salts containing two molecules of a monobasic acid or one molecule of a dibasic acid for each molecule of the alkaloid; and basic salts containing one molecule of monobasic acid for one molecule of the alkaloid.

Researches on Urobiline and Biliary Pigments.—E. Lépinois.—As a rule, the detection of urobiline is a relatively simple matter, but if biliary pigments are present at the same time it becomes more difficult. The author has simplified the old method by adding a known quantity of chloride of zinc, then dilute ammonia—insufficient to dissolve the precipitate formed. It is then filtered, and the alkaline liquid, which has become limpid by filtration, is fluorescent if urobiline is present. Besides being fluorescent, the solution also gives a spectrum in which either normal urobiline (urochrome) or the abnormal urobiline of febrile urines can be detected.

Characteristic Reaction of Cotton-seed Oil.—G. Halphen.—The author finds that the red colour which he noticed some time ago when a sulpho-carbonic solution of the zinc salts of the fatty acids of cotton-seed oil was evaporated down to dryness is not due to the oxide of zinc at all, but that the sulphide of carbon alone will bring about the reaction, though only after a long time—so long in fact that it cannot be usefully applied to analysis. In trying to hasten the reaction, it was found that if both the oil and the sulphide were dissolved in amyl alcohol, the phenomenon was considerably accelerated. The best method of procedure is to mix in a test-tube equal parts of the oil to be tested, amyl alcohol, and sulphide of carbon containing 1 per cent of sulphur in solution. This tube is plunged into boiling salt water, so that one-half or two-thirds is above the surface; after ten minutes or a quarter of an hour the colouration appears if cotton-seed oil is present.

Commercial Preparation of Oxygen.—MM. Dutremblay and Lugan.—The authors hope to re-introduce, with improved apparatus and methods, the process used by Tessié du Motay in 1867, but since abandoned. It consisted of decomposing, by means of superheated steam at 500°, the alkaline manganates, which are re-generated at the same temperature by a dry current of purified air. The apparatus required is an air-pump, a purifier (to remove the CO_2 and H_2O), a furnace for the manganates, an automatic distributor (to introduce alternately air and steam into the retort), and a gas holder furnished with a refrigerator to condense the watery vapour drawn in. The reactions take place at a low pressure, and as the oxygen is carried into the gas holder by means of the steam instead of through a pump, it is kept perfectly free from carbides. The old disadvantages, which consisted in the caking of the charge and the evaporation of soda, have been rectified and done away with, and there is no longer now any danger of explosions occurring.

No. 10.

On Phosphoglycerate of Lime.—MM. Adrian and Trillat.—The method of preparing phosphoglycerate of lime now generally in use consists in heating a mixture of phosphoric acid and glycerine, for six consecutive days, to a temperature of 100° to 110° ; the acidity is then neutralised with carbonate of lime and milk of lime, and the salt is finally precipitated with alcohol. Experiments have shown that the proportion of lime and phosphoric acid present is not constant, varying as much as from 19.5 to 24.5 for the former, and from 26.0 to 33.6 for the latter. Its solubility in distilled water at 25° varies from 4.05 per cent to 7.6 per cent. The residue left by the commercial substance after solution in water consists of sulphate and phosphate of lime, in proportions varying from 0.10 per cent to 7.5 per cent. A slight elevation of temperature in an aqueous solution suffices to precipitate phosphoglycerate of lime; the precipitation commences at 32° , is very abundant at 40° , and almost complete at 100° .

On the Reaction of Filter-paper.—L. Magnier de la Source.—The author finds that filter-paper always retains a small quantity of acid, no matter how thorough the washing with cold water may have been: it is for this reason that it acts as an acid in titrations of cream of tartar in boiling solution, as the boiling water extracts the last traces of hydrochloric acid.

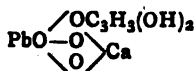
Qualitative Research on Traces of Alkaline Carbonates in presence of an Excess of Bicarbonates or of Borax.—Alex. Leys.—Already inserted in full.

On the Preparation of Distilled Waters.—M. Julliard.—The author does not agree with the report presented to the Pharmaceutical Society of Lyons by M. A. Lambert, on the revision of the Codex, that pharmacists are wrong in rejecting the preparation of aromatic waters by means of essences; his reason is, that in many cases distillation in itself causes an alteration of the molecular state of the essence.

On the Non-existence of an Intermediate Iodide of Mercury.—M. François.—Already inserted in full.

No. 11.

On Phosphoglycerate of Lime.—MM. Adrian and Trillat.—This paper is continued from the previous number. A sample of raw phosphoglycerate of lime gave on analysis 25 per cent of phosphoric acid and 22.5 of lime; after precipitation by heat, at a temperature of 70° , it gave 30 per cent of phosphoric acid and 23.25 per cent of lime. Contrary to the general opinion, phosphoglycerate of lime is not decomposed by prolonged heating at 100° . It has generally been described as an amorphous powder, but the authors have at last obtained it in the form of regular crystalline needles; when exposed to the air, however, these crystals gradually disintegrate, and form an amorphous powder. Purified phosphoglycerate of lime corresponds with the formula—



as given by Pelouze.

On the Origin of Artesian Water.—M. Lehache.—Not suitable for abstraction.

On Testing for Diastase from Barley.—P. Terrat.—The author shows that, in testing for barley diastase, the use of ordinary water is unsuitable, and may lead to the rejection of really good samples as being inferior: the chief cause of this difference in results is due to the alkalinity of the water, so small a quantity as 0.002 per cent of sodic hydrate reducing the titration of the diastase to 26 per cent of what it would be with a neutral water.

MISCELLANEOUS.

Australasian Association for the Advancement of Science.—The seventh meeting of this Association is now being held at Sydney, New South Wales, under the presidency of Prof. A. Liversidge, M.A., LL.D., F.R.S. The Association is divided into ten sections. Section B being devoted to Chemistry, under the presidency of T. C. Cloud, A.R.S.M., F.C.S., where papers will be read on "Metallurgical Methods in Use at Broken Hill," by G. H. Blakemore; "The Mineral Waters of Australasia," by G. Gray, F.C.S.; "Notes on the Constitution of Gluten," by F. B. Guthrie, F.C.S.; "The Characteristics of Australian and other Diamonds," by E. W. Streeter; &c. Besides these, there will be a large number of papers read in the other sections: those of "Ethnology and Anthropology" (Section F); and "Economic Science and Agriculture" (Section G), both having long and interesting programmes. The scientific side of the meeting will be agreeably leavened by entertainments and excursions; and we have no doubt but that this, the seventh meeting of the Association, will be as successful as the preceding ones.

McGill University, Montreal.—An additional Chair of Chemistry has been founded and endowed in this University by Mr. W. C. McDonald, who recently erected a new chemical building at a cost of 240,000 dollars. The same donor has provided an additional endowment of 50,000 dollars for the Faculty of Law, to the deanship of which Faculty, with the Chair of Roman Law, Mr. F. P. Walton, of the Scotch Bar, was recently appointed. Mr. McDonald has, moreover, supplemented the existing endowments associated with his name by a further gift of 200,000 dollars, to provide for any deficiency in income that may result from the fall in the rate of interest on investments.

On Silver Cyanamide, CN_2Ag .—Paul Lemoult.—Silver cyanamide is one of the most characteristic derivatives of cyanamide. It is formed by the reaction of an ammoniacal solution of NO_2Ag on an aqueous solution of cyanamide; it occurs as a yellow precipitate, but it is not easily obtained pure. To effect this, it must be suspended in water and acted on by an excess of HNO_3 in the cold. After filtration (the solution is never complete), the liquid is precipitated little by little with ammonia, and after repeating this several times the pure product may be obtained.—*Comptes Rendus*, cxxv., No. 20.

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FIG. 11. No. 81.

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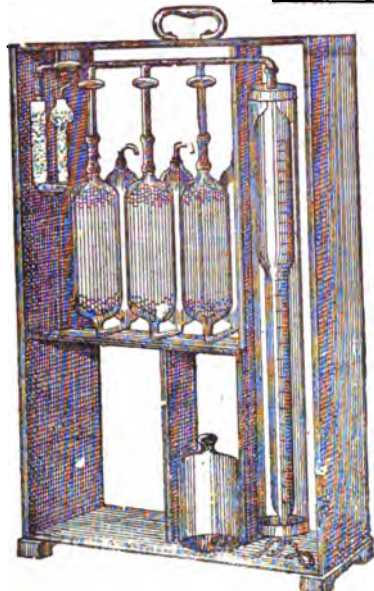
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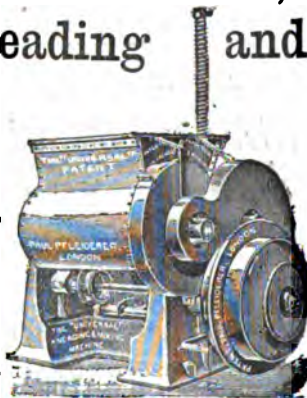
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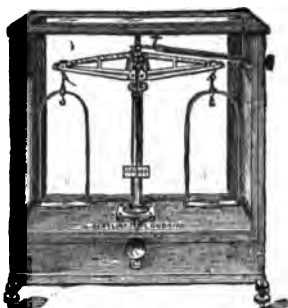
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CAMBRIDGE MASS.

SOME FURTHER DETERMINATIONS OF THE DIELECTRIC CONSTANTS OF ORGANIC BODIES AND ELECTROLYTES AT VERY LOW TEMPERATURES.*

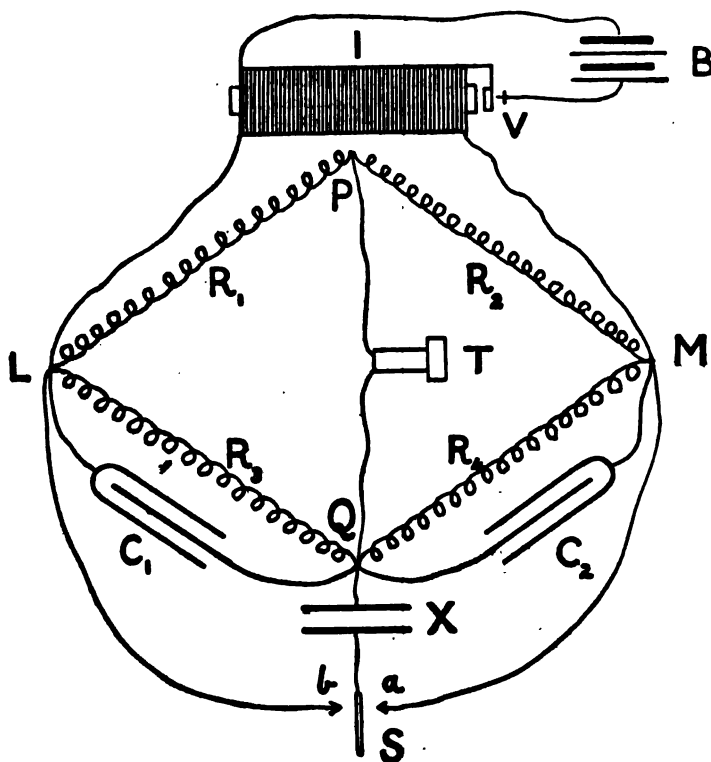
By JAMES DEWAR, M.A., LL.D., F.R.S., Fullerian Professor of Chemistry in the Royal Institution,

and
J. A. FLEMING, M.A., D.Sc., F.R.S., Professor of Electrical Engineering in University College, London.

In several previous communications (see Fleming and Dewar, *Roy. Soc. Proc.*, 1897, vol. lxi., pp. 2, 299, 316, 358, 368, and 381) we have described the investigations

pose the apparatus as arranged by Dr. Nernst which belongs to the Davy-Faraday Laboratory. The frequency of alternation employed was 350 or thereabouts, whereas in all our formerly described experiments with the galvanometer method it was 120.

The electrical details of the arrangement employed in Nernst's method are as follows:—A Wheatstone's bridge is formed (see diagram), two sides of which consist of variable resistances, R_1 , R_2 , which are usually liquid resistances contained in U-tubes. The other two sides of the bridge consist of two sliding condensers of variable capacity, C_1 , C_2 , which are shunted by adjustable liquid resistances, R_3 , R_4 . The bridge circuit contains a telephone, T , as detector. The alternating currents are furnished by an induction coil, i . An experimental condenser, X , the dielectric of which can be made to be the substance under examination, is connected, as shown in the diagram, so that it can be placed in parallel with either of the shunted condensers forming the third and fourth arms of the bridge. The process of measurement is then as follows:—The experimental condenser is placed in parallel, by the switch s , say, with the third arm of the bridge, and the shunt resistances are first adjusted, so that



made by us on the dielectric constants of various frozen organic bodies and electrolytes at very low temperatures. In these researches we employed a method for the measurement of the dielectric constant which consisted in charging and discharging a condenser, having the given body as dielectric, through a galvanometer 120 times in a second by means of a tuning-fork interrupter. During the past summer we have repeated some of these determinations, and used a different method of measurement and a rather higher frequency. In the experiments here described we have adopted Nernst's method for the measurement of dielectric constants, using for this pur-

the telephone gives a minimum of sound. The capacity of one sliding condenser, C_1 , is then varied until complete silence in the telephone in the bridge is obtained. The experimental condenser is next shifted over into parallel with the fourth arm, and the capacity of the same sliding condenser is again adjusted to produce silence in the telephone. The change in capacity thus made in the sliding condenser corresponding to the change in position of the experimental condenser is denoted by s . The experimental condenser then has its air dielectric replaced by a liquid of known dielectric constant D_0 , and the same process of change of its position effected. Let the variation of the adjustable sliding condenser then be denoted by S_0 . Finally the experimental condenser has its air

* A Paper read before the Royal Society, December 9th, 1897.

dielectric replaced by a liquid or frozen liquid of unknown dielectric constant D , and the process of change and adjustment a third time repeated. Let the variation of the sliding condenser in this third case be S . It is then very easy to show that the following relation holds good between D_0 , D , S_0 , S , and s , viz.:—

$$\frac{D_0 - 1}{S_0 - s} = \frac{D - 1}{S - s}$$

or —
$$D = \frac{D_0 - 1}{S_0 - s} (S - s) + 1.$$

In order to apply this method we employed absolute ethylic alcohol as the standard dielectric substance of known dielectric constant, and took as its dielectric constant at 15° C. the value 25.8 (according to Nernst), a number closely in agreement with all the best results by other observers.

The actual capacity of the experimental condenser with air as dielectric was very small, not being more than about 0.00001 of a microfarad. Hence in the above formula $D_0 = 25.8$.

The value of s was determined to be 1.33 and 1.38 in two experiments, and the mean 1.36 was taken as the value of s .

The following liquids were then examined by placing them in the experimental condenser and keeping them at ordinary temperatures, viz., 16° C. to 20° C.

1. Solution of potassic hydrate in water, 5 per cent solution.
2. Solution of rubidic hydrate in water, 5 per cent solution.
3. Amyl alcohol.
4. Ethylic ether.
5. Ethylic ether, pure and dry.
6. Ethylic alcohol.

The change in capacity of the sliding condenser when ethylic alcohol replaced the air in the experimental condenser was 16.7. Hence $S_0 = 16.7$ and $D_0 = 25.8$, also $s = 1.36$.

Therefore—

$$\frac{D_0 - 1}{S_0 - s} = \frac{24.8}{15.34} = 1.613.$$

The following table shows the observed values of s in the several cases when the above liquids were placed in the experimental condenser, and the corresponding calculated value of the dielectric constant D , where—

$$D = 1.613 \times (S - s) + 1.$$

TABLE I.—*Determinations of the Dielectric Constants of certain Liquids at Ordinary Temperatures (15° C.) by Nernst's Method. Frequency = 320.*

Substance.	S.	S-s.	$\frac{1.613}{(S-s)}$	Dielectric constant = D.
Ethylic alcohol (taken as the standard of comparison)	16.7	15.34	24.8	25.8 (assumed value)
Amyl alcohol	10.49	9.13	14.7	15.7 (calculated)
Ethylic ether	3.98	2.62	4.23	5.23 (calculated)
Pure dry ethylic ether	3.70	2.34	3.78	4.78 (calculated)

Hence by Nernst's method, assuming the dielectric constant of ethylic alcohol to be 25.8, we find that of amyl alcohol to be 15.7 and pure ethylic ether to be 4.78.

Nernst himself found amyl alcohol to be 16.0 and ethylic ether to be 4.25 at about this temperature. Hence our values are in fair agreement with his.

In the next place we cooled the experimental condenser down to the temperature -18.5° C. in liquid air, after filling it with one of the above six dielectric liquids, and we repeated all the above-described operations again. The results are collected in Table II.

TABLE II.—*Determinations of the Dielectric Constants of certain Frozen Liquids at the Temperature of Liquid Air by Nernst's Method. Frequency = 320.*

Substance.	S.	S-s.	$\frac{1.613}{(S-s)}$	Calculated dielectric constant = D.
Ethylic alcohol	2.68	1.32	2.13	3.13
Amyl alcohol	2.34	0.98	1.58	2.58
Ethylic ether	2.16	0.80	1.29	2.29
5 per cent solution of rubidic hydrate	2.94	1.58	2.55	3.55
5 per cent solution of potassic hydrate	5.15	3.79	6.12	7.12

The above values for the organic bodies are in close agreement with the results we obtained for the same substances by the galvanometer and switch method formerly used by us, as may be seen by a reference to Table III.

TABLE III.—*Comparison of the Determinations of certain Dielectric Constants made by Different Methods at the Temperature of Liquid Air.*

Substance.	By galvanometer and switch method. Frequency = 320. Dielectric constant.	By Nernst's bridge method with telephone. Frequency = 320. Dielectric constant.
Ethylic alcohol	3.11	3.13
Amyl alcohol	2.14	2.58
Ethylic ether	2.31	2.29
5 per cent solution (aqueous) of potassic hydrate	123.0	7.12
5 per cent solution (aqueous) of rubidic hydrate	81.6	3.55

The results collected in the above Table III. show that the two methods give practically identical values for the two alcohols and the ether, but very different value for the two frozen dilute hydrates.

An examination was then made of several other substances, and for this purpose another condenser was constructed, which consisted of a platinum crucible about 4 c.m. in diameter and 5 c.m. high. This crucible was fitted with an ebonite lid, through which passed a glass test-tube, in the interior of which was placed our platinum thermometer. Round the outside of the test-tube, platinum wire was closely wound, so as to form the opposed surface of a condenser in relation to the platinum crucible as the other surface. This platinum condenser could then be filled with any electrolyte or organic liquid and frozen in liquid air.

Owing to the very small actual capacity of this last experimental condenser, and especially that of the variable part of it in comparison with the capacity of the leads and connections, no very great accuracy of measurement was looked for or attained. The results, however, were sufficient to check the general accuracy of the experiment with similar substances by the galvanometer method. This platinum condenser was calibrated and used with the Nernst bridge, exactly as in the previous experiment.

With the experimental condenser empty, the change in capacity of the variable sliding condenser in the bridge arm was 1.50 on changing over the position of the experimental condenser. Hence $s = 1.50$.

When filled with ethylic alcohol ($D_0 = 25.8$) the change of capacity of the sliding condenser was 6.20. We have, therefore, $S_0 = 6.20$, $D_0 = 25.8$, $s = 1.50$.

Therefore—

$$\frac{D_0 - 1}{S_0 - s} = \frac{24.8}{4.70} = 5.27.$$

The experimental condenser was then filled with some liquid, either at ordinary temperature or frozen at a low temperature, and the bridge measurement made, and the reading S or the change of capacity of the sliding con-

denser observed, as before, when the experimental condenser had its position changed.

The following Table IV. gives the summary of the results obtained with several substances at various temperatures.

TABLE IV.—Measurement of the Dielectric Constants of various Substances at Different Temperatures by Nernst's Method. Frequency = 350.

$$S = 1.50, \frac{D_0 - 1}{S_0 - 1} = 5.27.$$

Substance.	S.	S-1.	$\frac{S-1}{S_0-1}$ (S=5.27)	Calculated dielectric constant = D.	Temperature in platinum degrees.
Ethyl ether	2.1	0.6	3.16	4.16	+15°
Glycerin	12.15	10.65	56.2	57.2	+30
Solution of ammonia (sp. gr. = 0.880) ..	4.5	3.0	15.8	16.8	-123 (?)
	5.6	4.1	21.6	22.6	-137
	13.5	12.0	63.0	64.0	-119
Distilled water or pure ice	0.2	0.5	2.6	3.6	-49
	11.0	9.5	50.0	51.0	-7
	14.3	12.8	67.0	68.0	+1
Oxide of copper in suspension in water	3.95	2.45	12.9	13.9	-61
	12.8	11.3	59.7	60.7	-41

The value found for the dielectric constant of water at +1° is rather low, but, as above mentioned, the smallness of the capacity of the experimental condenser prevented the results from being more than good indications of the order of the dielectric constant.

(To be continued).

THE PLASTER OF PARIS METHOD IN BLOWPIPE ANALYSIS.

By Professor W. W. ANDREWS.

In a paper published last October in the *Journal of the American Chemical Society*, entitled "Some Extensions of the Plaster of Paris Method in Blowpipe Analysis," the author gave some account of the development of this method since Dr. Eugene Haanel first proposed this new support. The new composition of the tablets, the new easily prepared and portable reagents, and the reactions they yield with the metals were there described. The author now gives some new applications of this support and some new reactions. The iodide coatings of the metals are shown.

The addition of boric acid to the calcium sulphate in the manufacture of the tablets so fortifies them that they form a substitute, not only for charcoal, but also for platinum wire and bone ash. They resist the action of the fluxes of borax and metaphosphoric acid, and instead of beads in platinum loops we may with advantage produce coloured glassy films on the surface of the white tablets. Oxidation and reduction take place very readily in these films. All degrees of saturation may be observed at once. The colour changes due to change of temperature may be more accurately observed on account of the slower rate of cooling.

In assay work, if a fragment of a tablet be heated to redness for a few seconds and then pulverised, we have a material which may be moulded into a smooth cupel which does not blister, and which very readily absorbs the lead oxide in cupellation.

Potassium sulphocyanate, metallic iodine, potassium cyanide, potassium sulphide, and potassium-cadmium cyanide are all easy to carry. With water they

easily dissolve, and therefore solutions may be prepared anywhere. In the laboratory more rapid and better work can be done with the solutions. Potassium sulphocyanate is not always kept by the druggists, and its preparation by crystallisation in the laboratory is tedious. The simplest way to prepare the iodine solution is to mix fragments of potassium cyanide and sulphur, the latter a little in excess of the molecular proportions, in a test-tube and fuse together, adding water while yet warm, and then adding metallic iodine to saturation. Dr. Wirt Tassin, of Washington, uses a solid reagent made by fusing iodine and the sulphocyanate together with a little sulphur, and then powdering. This, if stable, ought to prove very satisfactory.

Here is a very effective portable blowpipe lamp which cost less than two cents. It consists of an ordinary druggist's tin salve box, with a piece of tin bent to form a wick-holder. The cover is bulged so as to shut down over the wick. The fuel is paraffin wax or stearin. The flame is smokeless, very hot, and with great reducing power and free from sulphur. Once filled it burns for more than an hour. A test tube can be readily boiled over it. In private laboratories, and in schools in towns, where the electric light has supplanted gas, and affixed this little piece of simple apparatus should prove useful.

Some reactions, not hitherto published, are those obtained by using the tablets as infusible filters. Films are obtained, for which I propose the name "solution films," to distinguish them from the sublimation films.

If to a solution of a lead salt a little potassium sulphocyanate and iodine be added, a precipitate of lead iodide tends to form. When, however, no precipitate is visible, if a drop be let fall on a tablet instantly, a bright-yellow spot is seen. The delicacy of many tests may be greatly increased by making use of this property of the tablets. It seems to be somewhat catalytic. Not only one, but as many as fifty or one hundred drops may fall upon the same spot, each drop deepening the colouration.

The iodine solution shows a remarkable power of dissolving gold. Gold leaf dropped on its surface almost instantly dissolves. If the solution containing gold be dropped on a tablet and the spot touched with the blowpipe flame, a fine pink film appears. One drop of solution containing one part gold in thirty thousand will show a fine pink. Fifty drops will show gold present in one part in six hundred thousand, and one hundred drops one part in one million of solution. The test, therefore, may be made quantitative.

Platinum yields a slate-coloured film, chromium a film dark-green hot, and fine green cold. Copper yields a purple film, which, treated with sulphuric acid, disappears and darkens in oxidising flame. If a copper solution be dropped on a tablet and heated vapours of hydrobromic acid be blown over it, the purplish brown of cupric bromide will appear. This will reveal copper, when present, one part in two million parts of solution.

Iron gives a brownish film, which sulphuric acid turns to Venetian red, and other acids remove. A little metaphosphoric acid added to the solution will prevent the formation of the film. Cobalt yields a pink film, which becomes, on hydration, a beautiful blue, and more strongly heated a black, which a drop of strong acid potassium sulphate removes.

Nickel yields a pale green film, which, dehydrated, becomes a brownish yellow and then a black. Ammonium hydrate turns the solution a fine blue.

Nickel may be detected in the presence of cobalt. The solution will probably be colourless, unless iron be present, when it will be amethystine. If a drop of the iodine solution be added to the section of the tablet wetted by the metallic solution, and heated, the centre will be black, showing nickel, for in such circumstances the cobalt tends to leave a white centre, forming a black ring with sometimes a blue ring separating it from the centre, outside that a brownish yellow showing nickel, and further out a spreading blue showing cobalt. The

* Read before the British Association (Section B), Toronto Meeting, 1897.

salts experimented with were the nitrates and sulphates, and when cobalt was six times as abundant as the nickel, the reactions of the latter were well marked.

Manganese, vanadium, molybdenum, ruthenium, and osmium also yield solution films.

Potassium-cadmium cyanide has been found to be a reagent which affords a very delicate and ready test for sulphur even in the presence of selenium and tellurium.

Potassium sulphocyanate solution was added to a cadmium solution and dropped on a tablet and heated, and the scarlet of hot cadmium sulphide showed itself. Sixteen drops revealed the presence of cadmium in a $\frac{1}{100}$ normal solution, and this test was not interfered with by the presence of two hundred times as much zinc.

Zinc in the cobalt test responded with great delicacy, but aluminium gave no satisfactory results.

Another extension of this method is to the compounds of organic chemistry.

Carbon gives a sooty coating which metaphosphoric and sulphuric acids increase. Tars and asphalts give a black tinged with green.

The phenols with the same reagents yield a black edged with a pinkish red. Picric acid is the only exception found so far. It gives a yellow. The following were among the phenols treated—carbolic acid, pyrogallol, salicylic acid, oils of conine and wintergreen, Canada balsam, Burgundy pitch, resin, phenolphthalein, hydroquinone, and creolin. Many of the phenols when dropped on a tablet previously heated before the blowpipe show very brilliant and characteristic colours.

The paraffins yield no red, but heavy sooty, films.

HYSTERICAL CHEMISTRY.*

By Prof. H. CARRINGTON BOLTON, Ph.D.

(Concluded from p. 5).

GUSTAV MARQFOY is a writer on chemistry less fanciful than those cited, yet worthy of a place among hysterical chemists. In his "Loi des Equivalents et Théorie Nouvelle de la Chimie" (Paris, 1897) he admits the unity of matter, and reduces the number of undecomposable bodies to 63. The equivalents of these 63 simple bodies are prime numbers, forming a natural series from 1 to 300. The atom he regards as made of a nucleus and a plasma; the nucleus is the seat of energy. His view of the successive steps in the creation of the elements, in accordance with the arithmetical chain he discovered, is interesting, but not easily abstracted.

In this group of esoteric students the most picturesque personage is August Strindberg—a Swede by birth, a Frenchman by adoption, an Austrian by residence (temporary perhaps)—he is thus portrayed by one of his admirers:—Pale blue, dreamy, sympathetic eyes; a broad forehead, on which the fire of genius burns with brilliancy; slow of speech, with a marked Scandinavian accent, he attacks boldly the "routine and persecuting scientists;" a "superb man," disinterested, mild, invincible in perseverance, and seeking only the truth. Strindberg is a man of letters, being the author of successful romances and dramas,† and he claims to be a chemist, but his right to this claim can be better determined after analysing his pretensions.

Strindberg accepts the theories of the unity of matter, the self-evolution of elementary bodies, the transmutability of elements, and hylozoism; he even grants to matter the principles of heredity, memory, and love. His strength, however, lies in his peculiar method of demonstrating

that the chemical elements are compounds, and in discovering new formulæ for well-known substances. More proficient in manipulation than his Monist friends, he invokes results of laboratory work to confirm his demonstrations. With admirable courage he attacks problems in chemistry of herculean difficulty, and does not hesitate to undertake to disprove orthodox views of most fundamental facts, such as the composition of the atmosphere, the composition of water, and all the important phenomena depending thereon. Exhibiting considerable knowledge of the literature of chemistry, he is quick to discern in the earlier writings statements that he can turn to his advantage, ignoring the circumstance that the very statement on which he relies has often been shown to be erroneous by later authorities. He writes, for example, "Lampadius believed that carbon disulphide was composed of sulphur and hydrogen, had he said carbon and hydrogen I would bless his memory." Avicenna, Albertus Magnus, Paracelsus, Boerhaave, Berzelius, Bunsen, and Clève are, for Strindberg, authorities of equal weight. He uses on one page the very arguments which on another page he condemns when employed by "official" chemists, and he claims that the latter suppress the dogmas of hysterical chemistry to avoid the unpleasant necessity of revising theories of scientific chemistry. "About sixty years ago," he writes, "a French chemist decomposed chlorine and obtained 3 per cent of oxygen, but his discovery was buried to save appearances" (*Hortus Merlini*).

Strindberg denies the idea of an undecomposable body. "Is there truly a chemist," he asks, "who believes that iron dissolved in sulphuric acid is not decomposed?" He uses current chemical terms with changed meanings: precipitation signifies reconstruction; solution signifies decomposition; and combustion, synthesis—an original nomenclature that obscures his writings.

"The atom is an hypothesis,
An hypothesis has no weight, therefore
Atomic weight is a non-entity."

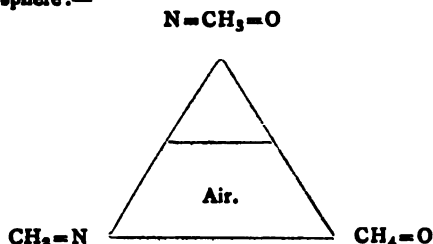
In spite of this remarkable syllogism, with which Strindberg opens one of his essays, he makes most of his discoveries (?) by juggling with the numbers representing these very atomic weights. When the molecular weight of a compound happens to be equal to the atomic weight of an element, or to the molecular weight of some other compound, he assumes that this accidental correspondence involves close relationship—if not identity—of substance, and he audaciously substitutes one formula for the other. In his "Introduction à une Chimie Unitaire" (Paris, 1895), Strindberg opens the topic with the motto "*Omnia in omnibus, omne omne est*," and proceeds thus:—"Bodies exist only in numbers and qualities; bodies are neither simple nor compound. Chemical formulæ are merely methods of expressing the manner in which bodies differ and correspond."

"That which is called hydrogen is neither a simple body nor a compound body; it is matter having the condensation 1. Carbon can be formulated thus: $H_{12}=C$; oxygen in the same way, H_{16} or $CH_4=16=O$; nitrogen likewise, H_{14} or $CH_2=14=N$; though nitrogen in combination may have the formula $N_2=28$." To the molecule of nitrogen he assigns the formula CO, because its molecular weight is 28. His method of reasoning is as peculiar as his choice of formulæ:—"By assigning to nitrogen the formula CH_2 or C_2H_4 , and to carbonic acid the formula N_2O , a whole series of phenomena hitherto obscure are explained. Thus the rôle of nitrogen in animal and vegetable economy: the nutrition of animals by carbohydrates, and the great excess of nitrogenous matter secreted; the miraculous way in which plants nourished with water and air produce carbon and albumenoids without absorption of nitrogen and without sufficient carbonic acid, the latter being only an impurity of the atmosphere (4 parts of CO_2 in 10,000 of air); the transformation of starch into albumenoids; and,

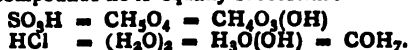
* Read to the Washington Section of the American Chemical Society, November 11th, 1897.

† His "Sensations d'un Détraqué" was published in *Figaro*. His drama "Magda" was acted at the Renaissance, Paris, by Sarah Bernhardt.

lastly, the origin of the creation by water and air, even Pasteurised, at the beginning of time." According to this ingenious philosopher, "that which is called air is neither a mixture nor a combination; it is neither oxygen nor nitrogen; it is both or neither. Since the atomic weight of carbon varies from 11 to 12 (?), nitrogen may be indicated by CH_2 or CH_3 ; since oxygen varies from 15 to 16, it may be represented by CH_3 or CH_4 ; therefore, in the conditions in which oxygen appears identical with nitrogen, both are represented by CH_3 . Air is therefore CH_3 , which signifies oxygen and nitrogen." Having thus established a novel formula for air, he proceeds to discuss on that basis its function in combustion, oxidation, and respiration: reverting to the alchemical symbol for air, Δ , he gives the following lucid diagram, which summarises the knowledge of hysterical chemists concerning the atmosphere:—



Strindberg takes up the chief elementary bodies and treats them in like manner; to chlorine he gives the formula $(\text{OH})_2$ or $2\text{H}_2\text{O}$, and this, he says, explains the presence of chlorine in sea-water. With other well-known compounds he is equally successful:—



And by applying his method to metals he secures some startling results:—

Iron	=	$(\text{C}_2\text{H}_4)_2$	=	56	
Manganese	=	$(\text{C}_2\text{H}_3)_2$	=	55	
Zinc	=	$(\text{CH}_4\text{O})_2$	=	64	
Copper	=	$(\text{CH}_4\text{O})_2$	=	63	
Copper	=	$\text{C}_2\text{H}_3\text{Cl}$	=	63	
Silver	=	$\text{C}_2\text{H}_5\text{Br}$	=	107	
Gold	=	Fe_2Si_3	=	197	[196]
Gold	=	Fe_3S	=	197	[200]
Sodium	=	C_2	=	24	(23)
Calcium	=	C_2H_4	=	40	
Nickel	=	$\text{C}_2\text{H}_5\text{P}$	=	58	
Cobalt	=	$\text{C}_2\text{H}_5\text{S}$	=	59	

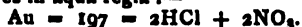
Of the two latter formulæ Strindberg remarks:—"Cobalt and nickel have always been suspected to be compounds, and I believe I have solved the problem of their enigmatical atomic weights."

The fact that the above equations are not always true, that many of the arithmetical calculations are inaccurate, that a few of the substances formulated are purely imaginary, and that the atomic weights occasionally differ by a whole number from the standard, does not at all disturb the equanimity of this Monist philosopher; when discrepancies of any kind are pointed out to him, he simply responds:—"Chemists always find impurities in the flasks of others, and never in their own."

"*Similia similibus dissolvuntur; similia similia appetunt*" is another maxim that this author illustrates as follows:—Copper dissolves best in concentrated nitric acid because they have the same weights:—



Gold dissolves in aqua regia:—



Zinc dissolves best in sulphuric acid:—



But $\text{CH}_4\text{O} = 8$, and



The chlorides in general are soluble in water, because of the constitution of chlorine, which is $(\text{OH})_2$ or O_2H_2 .

In his "Hortus Merlini" (first letter) Strindberg undertakes to demonstrate the compound nature of sulphur, claiming for it a constitution analogous to a fossil resin. After a detailed study of its external properties and its physical changes when heated, he recounts some original experiments, such as reduction of melted sulphur by the green twig of a chestnut tree (!), and burning sulphur in carbonic acid, which, he says, yields a deposit smelling like pepper; he claims that spectroscopic analysis of hydrogen flame shows the spectrum of sulphur; these and a score of equally convincing data go to prove that sulphur is a compound of C, H, and O. He ingeniously concludes thus:—"To require me to give a definite formula for sulphur would be indiscreet." Elsewhere he writes of "selenium the parent of sulphur."

Perhaps the best example of Strindberg's peculiar line of reasoning is found in an article entitled "La Synthèse de l'Iode," published in *L'Hyper-Chimie* for January, 1897:—"Sea-water contains not a trace of iodine, and consequently the algæ must produce it by synthesis. But living algæ yield no iodine, and do not yet contain it. The ashes of algæ, however, furnish iodine through the intervention of reagents; so it is evident that iodine is formed by a commutation of the crude materials in algæ and reagents. The old method of extracting iodine was by treating the mother-liquors with sulphuric acid and manganese dioxide; wherefore manganese? Because there exists an occult correspondence between the algæ, iodine, and manganese, which manifests itself in the colours they yield. Algæ occur of brown, greenish blue, reddish purple, and violet colours, and these same colours appear in iodine and in compounds of manganese." Strindberg then points out that there is a constant difference of 46 between the atomic weights of Cl, Br, and I; and having previously demonstrated that the correct formula for Cl is $(\text{OH})_2$, he then seeks for the body which has the weight 46. Finding a choice of three substances, viz.,—

Ethyl alcohol	=	$\text{C}_2\text{H}_6\text{O}$	=	46
Formic acid	=	CH_2O_2	=	46
Nitrogen peroxide	=	NO_2	=	46,

he selects formic acid, because the starch in the algæ is changed to sugar by sulphuric acid, and this sugar oxidised by manganese dioxide yields formic acid, so that he gets the following formula for iodine:—



Later in this amazing essay he regards iodine as a derivative of benzene,—



as experimental proof of this correspondence he states that oxyhydroquinone heated with alcohol gives out a vapour that colours starch test-paper blue and colours bread yellow, just as iodine does.

He offers to manufacturers of iodine practical suggestions based on these vagaries, which are too absurd to transcribe.

Denying the elementary character of the metals, Strindberg naturally believes in transmutation, and claims to have accomplished the "great work" in more than one instance. By dissolving pure copper in linseed oil, he finds in the product by analytical tests only nickel, and he claims to have obtained gold from lead and sulphur. He appeals to the transforming power of growing plants as proof of transmutation in Nature, and he ridicules the methods of official analytical chemists who examine the ashes of plants to ascertain the nature of the plants:—"Does anyone make an inventory of the treasures of a palace by setting fire to the four corners of a Raphael or a Rembrandt, and does he determine their value by the remaining traces of cinnabar, white lead, and chrome yellow?"

This convincing method of reasoning he employs in another connection:—"People ask me if I can make gold, and I reply, 'To draw the genealogical chart of the ancestors of a cat, I do not need to know how to make a cat!'"

Though their theories and methods are indisputably original, hysterical chemists, so far as appears from their writings, have contributed nothing whatever to advance the science they profess to admire. If anyone of their number appears to be a genius, it must be remembered that "a genius is a man who is almost a fool." Moreover, paraphrasing Rabelais,—

"The world of fools bath such a store
That he who would not see an ass
Must bide at home, bolt his door,
And break his looking-glass!"

CONTRIBUTION TO THE STUDY OF THE NITRIFICATION OF SOILS.

By TH. SCHLÖSING, Jun.

NITRIFICATION, and the microbial combustions of organic matters generally, are, as a rule, more active in rich soils than in light ones. This is a recognised fact, concerning which M. Muntz has given definite examples, and with which practical men agree, by saying that strong soils conserve manure. It is generally explained by the greater facility with which the air penetrates light soils.

In most cases, however, it is not the air—I do not say in all cases (see the work of M. Dehérain on this subject, *Comptes Rendus*, vol. cxxi.)—but the absence in strong soils of the water which is necessary for the complete combustion to go on.

Normally, when water is not present in excessive quantities, it does not fill up the interstices between the solid particles, but is spread over the surface of these particles in thin layers, which form a uniform covering as a whole. For an equal proportion of moisture in an earth, the thickness of these layers diminishes as the fineness of the particles increases, because of the increasing total surface of the latter. In a soil containing 10 per cent of water and a good proportion of clay (a substance composed of particles in a very fine state of division) this fineness may become so extreme as to seriously interfere with the nourishment of the microbes.

I now give a few experiments relating to the nitrification of ammonia, in which I have endeavoured to determine the influence of the thickness of the layers of water with which the particles of the soil are coated. I have made artificial soils from the following ingredients, in which the mixture was made as homogeneous as possible:—Quartz sand, from Bonnevault, in which the grains had an average diameter of $\frac{1}{2}$ m.m.; clay, from Vauves, the particles of which had an average diameter of $1\ \mu$; chalk, from Meudon; and water holding in solution a known quantity of sulphate of ammonia, and of which a part had been shaken up with a little earth, so as to be charged with nitrifying microbes, and then decanted. The mixtures were placed in separate flasks, large enough for there always to be an abundant supply of oxygen, and kept from such variations of temperature as would cause loss of moisture by evaporation. The nitric acid formed was eventually estimated.

Each lot, at the commencement, contained 50 m.grms. of sulphate of ammonia = 40.9 m.grms. N_2O_5 .

FIRST SERIES.—January 14 to March 27. Temp. 26–27°.

Composition of the soils.	I.	II.	III.	IV.	V.
	Grms.	Grms.	Grms.	Grms.	Grms.
Sand	100	90	80	75	70
Clay	0	10	20	25	30
Chalk	0.5	0.5	0.5	0.5	0.5
Water	10	10	10	10	10

N_2O_5 found (m.grms.)	34.1	38.5	36.2	23.5	4.23
N nitrified (per cent)	83	94	89	56	10

The same experiments were repeated, but the solid particles were first of all sterilised, and, as a check, the unnitrified ammonia was also estimated.

Each lot, at the commencement, contained 100 m.grms. of sulphate of ammonia = 81.8 m.grms. N_2O_5 .

SECOND SERIES.—July 17 to November 1. At the Temperature of the Laboratory.

Composition of the soils.	VI.	VII.	VIII.	IX.	X.	XI.
	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.
Sand	100	90	85	80	75	70
Clay	0	10	15	20	25	30
Chalk	1	1	1	1	1	1
Water	9.5	9.5	9.5	9.5	9.5	9.5

N_2O_5 found (m.grms.)	51.2	54.1	77.1	81.9	17	2.2
N nitrified (per cent)	63	66	94	100	21	2.7

The results of these two series are in accord. Complete, or almost complete, nitrification takes place in numbers II., III., VIII., and IX; decidedly weaker nitrification takes place in numbers IV., V., X., and XI. In other words, those containing 25 per cent or more of clay, and representing relatively strong soils, have undergone but little nitrification, while those corresponding to light soils have nitrified very well.

In numbers I. and VI. the nitrification was very incomplete, and the presence of nitrous acid was detected. It is probable that the proportion 9.5 or 10 per cent of water in pure sand was too much; the water evidently occupied too much of the interstitial spaces, and thus the aëration was insufficient.

If the nitrification was interfered with in numbers IV., V., X., and XI., can it be attributed to a want of air throughout the soils? No; and for this reason: that the soils, by the very method of their preparation, were thoroughly disintegrated, and in no way caked, but quite permeable; anyone who saw them could never believe that the aëration was insufficient. But the best answer to the question may be found through the following experiments, in which only the proportion of water was varied.

At the commencement, each lot contained 66.6 m.grms. of sulphate of ammonia = 54.5 m.grms. N_2O_5 .

THIRD SERIES.—April 1 to June 12. Temperature 26°

Composition of the soils.	XII.	XIII.	XIV.	XV.
Solid matters sterilised.	Grms.	Grms.	Grms.	Grms.
Sand	70	70	70	70
Clay	30	30	30	30
Chalk	0.5	0.5	0.5	0.5
Water	10.6	11.5	13.2	14

N_2O_5 found (m.grms.)	43.6	55.9	53.9	53.2
N nitrified (per cent)	80	100	100	100

We see here that by slightly increasing the amount of water—from 9.5 to 11.5 per cent—we have made the nitrification of the ammonia complete, where it was only partial. Now, this addition of water has rather diminished than increased the aëration, the liquid added occupying a part of the interstitial spaces formerly empty. It was therefore water, and not air, that was wanting in the soils containing 25 per cent and more of clay.

The strong soils of fields are often in this condition, that, though not wanting more air, they are short of water, so that nitrification and other microbial combustions are very much retarded; and it is for this reason that organic matters are consumed less quickly than in sands and coarser soils, where less water suffices.

Now, how is it that the want of water acts on the microbial combustions in soils? Let it be noticed that in the experiments quoted above (first and second series) its effect was distinctly shown in passing from soils containing 20 per cent of clay to those containing 25 per cent, and inversely that it was sufficient to add to the

soils very little water (third series) for its effect to be counteracted.

Between these two classes of soil—one nitrifying exceedingly well, the other badly—we can see no difference except in the thickness of the layers of water covering the particles; but even this difference was not great—in the one case 0.15μ , and in the other case 0.13μ (according to a rough calculation made with the object of getting some idea of the amount of difference). Thus, by increasing the proportion of clay only very slightly—that is to say, by diminishing the thickness of the layer of water by very little, we have considerably reduced the working capacity of the microbes, and we have restored this power by increasing the thickness again by even a very small amount. May we not therefore presume that, in diminishing this thickness beyond a certain point, we have reached a limit beyond which capillary attraction acts on the water and the dissolved principles with more force than the osmose, which causes the waters and the matters in solution to penetrate the microbial cells? The microbes have thus become unable to draw their nourishment from the soil. In any case it is interesting to see that so slight a variation in the thickness of the layer of water can have such a retarding action on the work of the microbes.—*Comptes Rendus*, cxv., No. 21.

ON A NEW METHOD FOR THE ATTACK OF PLATINUM. THE PREPARATION OF BROMO-PLATINATES OF AMMONIUM AND OF POTASSIUM.

By GEORGE MÉKÉR.

PLATINUM is considered to be unattackable to a large extent, while some saline mixtures which are looked upon as inactive attack it with great readiness.

Platinum, even when in a very fine state of division, resists the action of melted sulphate of ammonium; the alkaline chlorides and bromides affect it only to an inconsiderable degree at temperatures between 250° and 350° ; but, on the contrary, in the event of using a mixture of these salts, and above all with sulphate and bromide of ammonium, or what comes to the same thing, sulphate of ammonium and bromide of potassium the corrosion of the metal is very rapid.

If we heat such a mixture in an ordinary platinum crucible for some few instants, we can see that the surface where contact takes place becomes bright red, while the metal undergoes a corrosion similar to the well-known appearance of etched iron.

I have proved that the red salt thus formed is bromoplatinate of ammonium.

The only mention of the preparation of this salt up to the present time has been by Topsoë in 1868. This preparation consisted in the combination of bromide of ammonium with the tetrabromide of platinum. The latter salt was difficult to obtain, and I have endeavoured to use the method of the direct attack of platinum for the purpose of obtaining the bromoplatinate of ammonium with as little trouble as possible.

For this purpose we slowly melt in a porcelain crucible six parts of ordinary sulphate of ammonium, then, when the salt is melted and the temperature at about 300° , we add, while stirring, one part of bromide of ammonium, or for greater simplicity powdered bromide of potassium with which has been incorporated, by grinding together, platinum black, or even finely divided metallic platinum.

We must keep the whole stirring at about 330° , and it will quickly become bright red, and froth considerably, while at the same time white vapours of bromide and sulphide of ammonium will be given off. After heating for ten or fifteen minutes the mass is poured out and dissolved in the smallest possible quantity of water and

filtered. On the filter there remains a vermillion-red precipitate, while the solution will not contain any sensible amount of platinum; the salt formed being either insoluble or almost insoluble in ammoniacal salts. The precipitate is washed with a little cold water until the latter begins to be slightly coloured; the receptacle is then changed, and the filter washed with boiling water. The bromoplatinate goes into solution, and there remains a slight residue of undissolved platinum. The aqueous solution of the bromoplatinate, on being concentrated and cooled, deposits crystals of a bright crimson-red colour of great brilliancy, soluble in about two hundred times their weight of water.

The analysis of these crystals shows that they consist of pure bromoplatinate of ammonium.

	I.	II.	Theory per cent.	Found per cent. Average.
Substance ..	0.346	0.242	—	—
Platinum (by calcination)	0.095	0.0665	27.32	27.24
Substance ..	0.348	0.4425	—	—
Platinum (by magnesium)	0.097	0.123	27.32	27.68
Bromine ..	—	—	67.60	66.96
Nitrogen ..	—	—	3.94	3.85

The estimation of the platinum was carried out by two different methods; by direct calcination, and by precipitation by means of magnesium. In the latter case the figures obtained were always a trifle high, because of the traces of impurities contained in the magnesium. The bromine was estimated in the mother-liquors, from the precipitation of the platinum in the state of bromide of silver.

As in the case of the chloroplatinate, potash does not displace all the ammonium from the bromoplatinate; it is only by precipitating the platinum by magnesium that the magnesia formed by the action of the boiling water completely liberates the ammonia and makes the estimation possible.

By the method which has just been described, bromoplatinate of ammonium can be prepared in abundance with the greatest ease; but at the same time it must be borne in mind that, under the same conditions in other respects, the platinum is transformed only in proportion to the state of fineness in which it exists. In the state of sponge, or of very fine platinum black which has not been rendered metallic by rubbing, about two-thirds of the metal can be converted into the salt; the remainder, enveloped in a coating of crystals of bromoplatinate insoluble in the liquid, is not attacked. When using thin plates or filings it is advisable to rub the bottom and sides of the bath of the molten material so as to keep presenting new surfaces for the reaction. It is worthy of remark that the crystals of bromoplatinate deposited from a warm solution on cooling, take the form of cubo-octahedra, on which the octahedric facets are well developed, while those deposited by the spontaneous evaporation of a cold solution much more nearly resemble the cube.

By evaporation in the open air, we obtain, after a few months, cubical crystals of 2 m.m. to 2.5 m.m. in length, and occasionally possessing slight octahedric modifications. If we attempt to replace the bromide of ammonium or potassium by the chloride, we do not obtain a definite reaction; there is certainly a small quantity of chloroplatinate of ammonium formed, but there are at the same time ammoniacal derivatives, formed by the action of the products of decomposition of the sulphate of ammonium on the chloroplatinate, which appears from this to be less stable than the bromo-salt described above.

The substitution of an iodide for the bromide has not led to the formation of an iodoplatinate; the iodine being immediately displaced by the acid action of the melted sulphate and the intervening action of the air.

If the sulphate of ammonium (which when hot is really equivalent to a bi-sulphate) is replaced by bisulphate of potassium, we obtain a bromoplatinate of potassium; but the return is bad, the greater part of the bromine going off in the state of hydrobromic acid and free bromine. Further, the bromoplatinate of potassium is slightly soluble in salts of potassium, which renders the separation difficult, and it is no good attempting to make the salt insoluble by means of sulphate of ammonium, this latter having the curious property of acting on the bromoplatinate of potassium, and converting it into an ammonium salt. The mixture of bisulphate and chloride of potassium gives potassic chloroplatinate, but the operation is not practicable, and further, it is of no particular interest.

The reaction of the mixture of sulphates and bromide of ammonium or potassium on platinum is well defined and practicable; it has, further, a definite, specific character.—*Comptes Rendus*, cxv., No. 24, 1897.

ON PHOSPHOROUS OXIDE.

By A. BESSON.

I RECENTLY described the action which takes place in the cold between PCl_3 and crystallised PO_3H_3 ,—viz., disengagement of HCl and the formation of a syrupy liquid of an ill-defined composition, but which still remains stable when in contact with an excess of PCl_3 .

When warmed the reaction is more complete, and leads to the formation of a reddish yellow solid body, which is nothing else than phosphorous oxide, P_2O_3 , which I have already described in a previous communication as resulting from the reaction between PH_4Br and POCl_3 .

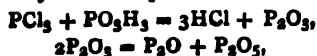
If a concentrated solution of PO_3H_3 , charged with an excess of PCl_3 , be heated on a water-bath at about 100° , in a flask furnished with a vertical condenser, there is a continuous disengagement of HCl , and the formation in the mass of the solution of the PO_3H_3 , which gradually thickens, of a solid body, bright yellow when in small particles and reddish yellow when in more considerable quantity. By continuing the operation, the syrupy mass becomes so thick that it is necessary to keep it constantly stirring, in order to renew the surfaces of contact. If, finally, we continue the reaction until cold, a solid, white, pulverulent body is formed, having the appearance of P_2O_5 . When the reaction is complete, and after cooling, we add water carefully, as its addition is accompanied with a considerable disengagement of heat, and sometimes by a sound analogous to that which accompanies the hydration of P_2O_5 .

The aqueous solution holds the reddish solid body in suspension; this is filtered off, washed with cold water, and dried *in vacuo* in the cold: this is the phosphorous oxide, P_2O_3 , which is by this means obtained in a very pure state.

The analysis of two separate samples from different operations gave the following results:—

P per cent 79.37 and 80.06
P theoretically 79.49

The mechanism of the reactions may be conceived to be as follows:— PCl_3 reacting on PO_3H_3 would give P_2O_5 , which would eventually decompose into P_2O and P_2O_5 ; this idea is in conformity with observations, and can be demonstrated by the two equations—



and it is to the formation of this latter body that the appearance, at the end of the reaction, of a solid white body is due, and also the disengagement of the greater part of the heat when water is added. It becomes therefore of importance to operate at as low a temperature as possible.

Phosphorous oxide is also formed by the direct oxidation of phosphorus under the following circumstances:—

A current of perfectly dry air is passed slowly through a solution of phosphorus in tetrachloride of carbon, the solution being protected from the light; chloride of carbon saturated with phosphorus at 30° to 40° might be used, and the oxidation carried on in the cold or in a tepid water-bath.

A yellowish white flocculent deposit soon appears, which soon thickens the solution. The liquid is then driven off over the water-bath, in a current of carbonic acid; the residue is washed with warm sulphide of carbon, of which the final traces are driven off in a current of CO_2 .

It is then carefully treated with water; part of the material is dissolved with disengagement of heat, the whole is thrown on a filter and a yellow matter is left, which is washed first with warm water and then with cold, and afterwards dried *in vacuo*. The material thus separated is nothing else than phosphorous oxide, as is shown by the following analysis:—

P per cent 78.85 and 79.10
P theoretically 79.49

It has all the chemical and physical characteristics of, and behaves quite in the same way as, the body previously described. In fine, phosphorous oxide, P_2O_3 , forms the major part of the impure product called *red oxide* or *yellow oxide*, and which contains either amorphous phosphorus or solid phosphide of hydrogen.—*Comptes Rendus*, vol. cxv., No. 24, 1897.

A REVISION OF THE ATOMIC WEIGHT OF COBALT.*

FIRST PAPER.—THE ANALYSIS OF COBALTOUS BROMIDE.

By THEODORE WILLIAM RICHARDS
and
GREGORY PAUL BAXTER.

Introduction.

SIMULTANEOUSLY with the work described in a previous paper ("A Revision of the Atomic Weight of Nickel," *Proc. Amer. Acad.*, xxxiii., No. 7; *CHEMICAL NEWS*, lxxvi., p. 284) a similar investigation of cobalt was in progress. Earlier work upon this metal had led to results quite as incapable of ready interpretation as those concerning nickel; even very recent work upon the atomic weight of cobalt allows a range of much over a unit (from 58.78 to 60.1) as the possible field within which the truth may lie. Professor Clarke has given a full summary of all the available data upon this subject in his recent work (*Smithsonian Miscellaneous Collections*, "Constants of Nature," Part V., p. 291) upon the Re-calculation of the Atomic Weights, and for references and details the reader is referred to this useful volume. A chronological list is reprinted here merely to show how urgent is the need for further labour.

Earlier Work on Cobalt. (O = 16).

			Atomic weight.
1826	Rothoff ..	$\text{CoO} : 2\text{AgCl} ..$	58.50
1857	Schneider ..	From cobaltous oxalate	60.00
1858	Marignac ..	$\text{CoSO}_4 : \text{CoO} ..$	58.76
1858	" ..	$\text{CoCl}_2 : 2\text{Ag} ..$	58.85
1860	Dumas ..	$\text{CoCl}_2 : 2\text{Ag} ..$	59.09
1863	Russell ..	$\text{CoO} : \text{Co} ..$	58.74
1867	" ..	$\text{Co} : \text{H}_2$ (measured) ..	59.08
1866	Sommaruga ..	$\text{Co}_2(\text{NH}_3)_{10}\text{Cl}_6 : 2\text{Co} ..$	59.95
1867	Winkler ..	$3\text{Co} : 2\text{Au} ..$	59.42

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

		Atomic weight.
1868	Weselsky ..	From complex cyanides 59.13
1871	Lee (under W. Gibbs) ..	From complex cyanides 59.20
1871	Lee (under W. Gibbs) ..	$\text{Co}_2(\text{NH}_3)_{10}\text{Cl}_6 : 2\text{Co}$.. 59.16
1886	Zimmermann	$\text{CoO} : \text{Co}$ 58.89
1889	Winkler	
1891	Remmler ..	$\text{CoO} : \text{Co}$ 58.80
1892	Schützenberger ..	$\text{CoO} : \text{Co}$ 60.12
1893	Winkler ..	$\text{Co} : 2\text{Ag} : 2\text{AgCl}$ 59.82
1894	" ..	$\text{Co} : \text{I}_2$ 59.52
1895	Hempel and Thiele ..	$\text{CoO} : \text{Co}$ 58.99
1895	Hempel and Thiele ..	$\text{Co} : \text{Cl}_2$ 58.78
1895	Hempel and Thiele ..	$\text{Co} : 2\text{AgCl}$ 58.91

From these absurdly varying results, Clarke (*Loc. cit.*) computes the value 58.932 as the most probable atomic weight, while Seubert (*Zeit. Anorg. Chem.*, xiii., 229) selects 59.6, and Ostwald (*"Lehrbuch,"* vol. i., p. 79) 59.0, as the true value. Of course, many of the figures above may be thrown out at once; but the evidence which remains after this first elimination is still too vague for anything like scientific certainty. It is not the purpose of the present work to discuss the accuracy of these bygone investigations; for such discussions are apt to be unconvincing. Inattention to a single matter of detail may easily overthrow all the value of quantitative labour; a post-mortem examination is not always able to detect

some than that of the corresponding salt of nickel. The bright green anhydrous crystals of cobaltous bromide are very much less easily decomposed than the brown nickel compound by water or oxygen at a high temperature, in spite of the fact that cobaltous bromide is very much the more hygroscopic and soluble in water when cold. In no case was a trace of an oxide of cobalt found among the crystals of bromide used for the analyses.

The first experiments were carried on in a hard glass tube heated by a "combustion" furnace, but as the high heat necessary to volatilize cobalt bromide played sad havoc with the glass, porcelain tubes, heated by means of a perforated Fletcher furnace (*Proc. Amer. Acad.*, xxxii., 63), were substituted. The arrangement was similar to that used in the research upon nickel. Into one end of a large porcelain tube (of which the internal diameter was 28 millimetres) was ground a glass connector fused to the apparatus for supplying a mixture of bromine vapour, hydrobromic acid, and nitrogen; while inside of the other end was "telescoped" a closely fitting smaller porcelain tube designed to serve as the receiver of the sublimed material. The boat containing the cobalt was of course placed in the hottest part of the furnace, and the open end of the inner tube was adjusted just beyond this point. At first the glass of the receiver tube was attacked by the hot bromine and bromides, and the first sample of sublimate was found to be contaminated by the decomposition products of the glass. Later the porcelain became covered with a brilliant-coloured coating of a cobalt glass which seemed to be permanent. The sublimate used in the analyses was taken more especially from the central portions of the sublimed masses, so that even if a small amount of alkali had been extracted from the porcelain it

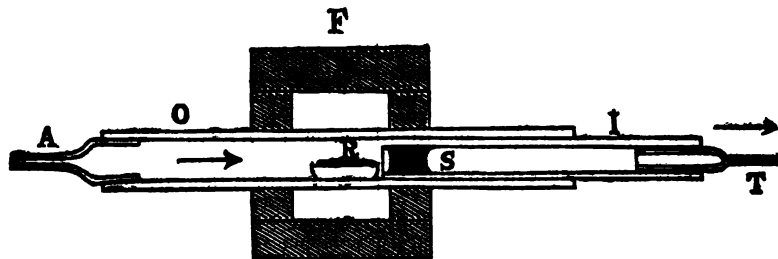


FIG. 1.—SECTION OF APPARATUS FOR SUBLIMATION.

A, Glass tube for admitting bromine vapour. O, Outer porcelain tube. I, Inner porcelain tube. T, Glass outlet tube. F, Perforated Fletcher furnace. B, Boat containing cobalt. S, Sublimed bromide.

the more subtle poisons. Our object is rather to accumulate a mass of data which we thoroughly understand, and then to interpret these data with regard to the probable chemical errors which may have crept into them. The present paper is only the first one of an investigation which we hope to continue until we have solved the problem.

The advantages to be gained by a simultaneous investigation of nickel and cobalt are obvious. Although the similarity of the two metals is usually overrated, it is still convenient to consider them together. Evidently much time may be saved in the preparation of materials if two similar researches are conducted side by side; and evidently the experience gained in one may be immediately helpful in the other. Fortunately, cobalt, like nickel, yields a bromide which is readily obtained anhydrous in a state of great purity, and this metal therefore joins the ranks of those elements whose atomic weights may be determined by reference to silver through the most satisfactory of all processes of precipitation.

The Preparation and Properties of Cobaltous Bromide.

All the cobaltous bromide used in this research was prepared by passing bromine vapour over hot spongy cobalt. The sublimation of the salt is much less trouble-

must have remained clinging to the walls of the tube. All the sublimate excepting the very first gave a wholly clear aqueous solution.

As has been already stated, anhydrous cobaltous bromide thus prepared consists of brilliant green crystalline plates, which quickly absorb water from the air to form the pink hydrated salt, and dissolve with great ease in cold water.

The specific gravity of anhydrous cobaltous bromide being unknown, the following experiments were made to determine it. (a) 2.0915 grms. of the salt which had been dried at about 200° displaced 0.3285 grm. of kerosene (dried over soda and re-distilled) at 25°. (b) 1.3965 grms. of the salt displaced 0.2186 grm. of kerosene. The kerosene, which was used in this case because cobaltous bromide was found to be slightly soluble in toluol, had at 25° a specific gravity of 0.7698 referred to water at 4°. Hence we have for the specific gravity sought, (a) 4.901, (b) 4.917. The mean of these two values is 4.909.

Since brass weights were employed, in order to correct the weights of cobaltous bromide found in air during the subsequent accurate analyses to that which would have been observed in a vacuum, 0.00010 grm. should be added for each grm. of this material. In the same way 0.00045 grm. must be added to every apparent grm. of argentic

bromide, and 0.000031 grm. must be subtracted from every apparent grm. of silver. These corrections are all applied below.

The balance used in this research was the one used in those upon copper, barium, strontium, zinc, and nickel: it needs no further description. The weights were of brass, gold plated; they were standardised with great care, and were used for no other work.

Purification of Materials.

Cobalt.—The doubt which has been thrown on the elementary character of cobalt by several experimenters led us to the application of unusual care in preparing the material used in this investigation. Pure cobalt was prepared by two entirely different methods, and a third sample was purified by both methods. The agreement of the results obtained from all of these samples leaves no doubt as to the uniformity of the material used.

In the first place about three hundred grms. of commercial cobalt chloride were dissolved in a litre of water. The removal of the copper group as sulphides was of course the first step, but on account of the difficulty of filtering out small quantities of these sulphides, the solution was not acidified before passing in sulphuretted hydrogen. In this way enough cobalt sulphide was precipitated to sweep the solution free from small amounts of a precipitate which could not be removed by filtration. After long standing the supernatant liquid was decanted through a filter, and as the precipitate was found to contain copper the solution was again fractionated with sulphuretted hydrogen. The sulphides from this precipitation gave no test for copper, hence it is fair to conclude that the filtrate was free from this metal. After the addition of a large amount of hydrochloric acid to this filtrate, and then ammoniac hydrate until the mixture was strongly alkaline, pure sulphuretted hydrogen in excess was passed into the solution. The precipitated sulphides were washed several times with water, and then digested with cold dilute hydrochloric acid. The precipitate was then washed free from iron by decantation. As the succeeding purification by means of potassic nitrite was in itself a separation from many other metals as well as from nickel, it was not thought necessary to re-precipitate the cobalt sulphide at this stage. Accordingly the precipitate was dissolved in aqua regia, and after dilution and filtration the solution was evaporated until the greater part of the excess of acid was driven off. Sodium hydrate was then added to the diluted solution in slight excess, and subsequently a large amount of acetic acid. Upon the addition of potassic nitrate all the cobalt was precipitated as the crystalline yellow double nitrite of potassium and cobalt. This precipitate was washed by decantation several times, and after its solution in strong hot hydrochloric acid the precipitation was repeated. The reagents used in this purification were especially tested for calcium; for only in the absence of calcium is the separation of cobalt from nickel complete by this method. The efficiency of the separation is shown by the fact that the filtrate from the first precipitation was distinctly green, owing to the nickel salts present, while the filtrate from the second precipitation after evaporation to small volume merely turned faintly brown on the addition of ammoniac sulphide. The double nitrite was again dissolved in hydrochloric acid, and the cobalt was partially freed from alkalis by precipitation as sulphide. The sulphide was then treated with an excess of nitric acid, and was heated upon the steam-bath until the resulting cobalt nitrate was free from every trace of chlorine.

In order to free the cobalt from alkalis it was next to be precipitated by electrolysis; and at first we hoped to convert the electrolytic film directly into bromide. However, on passing a galvanic current through the ammoniacal nitrate, an oxy-amine was precipitated in such quantities as to render the use of this electrolyte impossible. Experiments were then made with the ammoniacal sulphate in order to find the conditions most favourable for the de-

position of a bright coherent film. The negative electrode was a platinum dish, the positive electrode a flat spiral of platinum wire. The first attempts, which were made in a dish holding 250 c.c. and having an area of 150 square centimeters, with currents from 0.3 to 0.7 ampère, and with solutions of widely varying strengths, resulted in the deposition of dull black films. With a current strength of more than one ampère, however, the film was always comparatively bright. It was found impracticable to use this film directly for conversion into bromide on account of the difficulty of stripping the film from the dish. Accordingly, after being thoroughly washed, it was dissolved in the dish by nitric acid which had been distilled with a platinum condenser; and when the excess of acid had been driven off by evaporation, a slight excess of pure ammonia (made by distilling ordinary pure ammonia into pure water contained in a platinum dish) was added. The resulting green precipitate was washed several times by decantation with the purest water, filtered on a washed filter, dried in a steam oven, and finally, after separation from the filter paper, ignited to the black oxide in a platinum crucible. During the ignition this crucible was contained in a porcelain crucible which was heated by an alcohol lamp. Pure metallic cobalt was prepared from this oxide by reduction in a stream of ammonia gas, generated in an apparatus composed entirely of glass and dried by passage over stick-soda. Metallic cobalt is thus obtained in a spongy form, well adapted for its following treatment with bromine, and certainly free from all such impurities as alkalis and silica. The cobalt bromide, prepared from this metal in the manner already described, was labelled No. I.

The second method of preparing pure cobalt was by precipitation as one of its amines. The purpureo-chloride seemed best suited for the present purpose, both on account of the ease with which it can be prepared, and because of its comparative insolubility in cold ammoniacal or acid solutions. At first this compound was made by passing a current of air for some time through a solution containing cobaltous chloride, ammoniac chloride, and ammonia. A considerable quantity of purpureo-chloride was formed by this process, and from the solution, by acidification with hydrochloric acid and cooling, fully as much more was obtained. Later another method, capable of giving a much larger yield, was used. Into the strongly ammoniacal solution bromine was dropped slowly. A heavy precipitate resulted, and the solution upon acidification and cooling as above yielded the greater part of the cobalt it contained. Several lots of amines which had been prepared in one or the other of these ways were mixed and dissolved in hot ammonia. After filtering, the solution was acidified with hydrochloric acid, with the result that on cooling nearly all the cobalt was re-precipitated. The precipitate was washed by decantation with cold dilute hydrochloric acid, and the whole process of re-crystallisation repeated. This time the precipitate was collected on a pure filter and dried. After separation from the filter paper it was converted into the sulphates of cobalt and ammonium by heating on a sand bath with sulphuric acid until all chlorine or hydrochloric acid fumes had ceased to come off. From the solution of these sulphates metallic cobalt was prepared by electrolysis, solution in nitric acid, precipitation as hydrate, and reduction, in the manner already described. This preparation was designated as Sample II.

For the final analyses the cobalt was purified by a combination of all the before-mentioned methods. The carefully purified cobaltous nitrate from Sample I. was converted into a cobaltamine compound, and this salt was six times re-crystallised by solution in ammonia and precipitation with hydrochloric acid. Each time the precipitate was washed twice by decantation with hydrochloric acid, and after the second, fourth, and sixth dissolving the ammoniacal solution was filtered. The final precipitate was converted into spongy metallic cobalt in the usual manner. After sublimation part of the

bromide prepared from this cobalt was re-sublimed to see if this process had any influence on the salt. That which had been re-sublimed was labelled IV., the other III. The apparatus used for the sublimation has been already described.

Silver was prepared with great care, according to a method similar to that described in previous papers from this Laboratory (*Proc. Amer. Acad.*, xxxii., 62). The only improvement introduced was the removal by means of nitric acid of the surface of the buttons, which had been fused in a vacuum upon a boat of lime. This treatment is of course more thorough and satisfactory than Stas's method of digesting them with hydrochloric acid. The silver was subsequently treated with ammonia, thoroughly washed with water, and dried in a desiccator over soda-lime. One large piece was rolled out between clean steel rollers; and the foil, after having been cut into small strips, was cleansed as above described. These fragments made it possible to weigh out any desired amount of the metal.

Bromine was prepared in common with Mr. Cushman. For the method used the paper upon nickel should be consulted. Much experience has led to the conclusion that the method there described is the most satisfactory and convenient means of obtaining pure bromine, since it eliminates organic as well as inorganic impurities. In order to test the purity of the bromine and silver, 2.18679 grms. of silver (in vacuum) were dissolved in pure nitric acid and precipitated with a slight excess of ammoniac bromide which had been prepared by running the bromine in question into pure ammonia. The silver bromide was collected on a Gooch crucible and afterwards fused in a porcelain crucible. The fused bromide weighed 3.80679 in vacuum, whence the ratio of silver bromide to silver is 100.000 : 57.444. Mr. Cushman, experimenting upon the same sample of bromine, obtained the result 57.445; and since Stas's result was identical with these, there can be no doubt of the purity of our silver and bromine.

The sulphuric acid used in the drying towers was boiled for some time in order to increase its efficiency and to free it from volatile impurities. The phosphoric oxide was proved free from volatile compounds of phosphorus by passing a current of air over a considerable quantity of this substance in aqua regia for several hours. The aqua regia was subsequently found to contain no phosphorus. Distilled water was prepared with all the usual precautions necessary to free it from organic and non-volatile impurities, as well as from ammonia; nitric and hydrobromic acids were purified by repeated distillation with a platinum condenser; great care was taken to exclude dust, as well as the products of the combustion of illuminated gas, from the substances under treatment; platinum vessels were employed wherever it was possible to employ them; and many other precautions, often essential in order to prevent the complicated processes from introducing as much impurity as they removed, were applied. Alkali metals and silica, it will be seen, were among the impurities avoided as much as possible. For several pieces of platinum ware and other apparatus we are indebted to the Cyrus M. Warren Fund for research in Harvard University.

(To be continued).

NOTICES OF BOOKS.

Brewing Calculations: Gauging and Tables. By CLAUDE E. BATER, M.A., F.C.S. London: E. and F. N. Spon, Lim. New York: Spon and Chamberlain. 1897.

In the convenient little pocket-book now before us may be found tables connected with brewing in all its branches. The author has endeavoured to do without algebraic formulæ as far as possible, so as to make the book of use to junior officials. There are also other tables, more of general than of technical interest, and a good index.

Transactions of the Institution of Mining and Metallurgy. Sixth Session. Vol. V. London: Broad Street House, E.C. 1897.

The fifth number of this publication is a bulky volume of 346 pages, edited by Arthur C. Claudet, A.R.S.M. It contains a goodly number of papers on different mining and metallurgical subjects, which have been read before the Institution during the past year. It is of interest to note that more than half the papers and notes are devoted to the various branches of gold mining and recovery.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

Note.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Seances, de l'Academie des Sciences. Vol. cxxv., No. 23, December 6, 1897.

Contamination of Wells.—M. Duclaux.—The proof of pollution is afforded by the appearance in the water of certain wells at Cantal of two elements almost absent in the virgin waters of the same geological region, *i.e.*, lime and chlorine.

Dissociation and Polymerisation of Gases and Vapours. Presumed Dissociation of Chlorine at Elevated Temperatures.—A. Leduc.—V. Meyer has announced that the molecule of chlorine undergoes a very important dissociation at high temperatures. Subsequently Crafts, whilst admitting the existence of this dissociation, declares it less powerful. The figures of M. Meyer, though not agreeing well, seem to indicate that it is the same at 900° and 1200°. The figure of M. Crafts at 1400° indicates an important dissociation.

Electric Conductivity of Discontinuous Conductive Substances with reference to Telegraphy without Wires.—E. Bronly.—This paper will be inserted at the earliest opportunity.

Transformation of the X Rays of Metals.—G. Sagnac.—The author shows that the X rays are diffused over polished metals without regular appreciable reflection. The rectilinear propagation, without any sensible diffraction of the secondary rays from metallic surfaces, can be easily demonstrated by receiving them on a photographic plate; these rays are also non-refrangible and are not reflected to any extent.

Some Novel Facts observed in Crookes Tubes.—Virgilio Machado.—If we pass the electric discharge of a Ruhmkorff coil into a Crookes tube a variety of interesting facts are observed. For instance, the shadow of an aluminium cross appears at the anti-cathodic part of the tube as well as in the ordinary manner. When the tube is brought near a magnet these two shadows separate, but the displacement of the secondary shadow is greater than that of the principal one.

Accidental Causes of Irreversibility in Chemical Reactions.—A. Colson.—The author has established that the mutual displacement of two acids—the one free and the other salified—is a reversible phenomenon governed by the laws of heterogeneous dissociation.

Existence of a Cuprous Sulphate.—A. Joannis.—Experiments indicate that cuprous sulphate exists in combination with both carbonic oxide and ammonia.

Elementary Unity of the Substance known as Cerium.—M. Wyruboff and A. Verneuil.—A controversial paper on the elementary character of cerium.

Ammonium Aldehyde.—Marcel Delépine.—A Thermo-chemical paper on the representation of C_2H_7NO , *i.e.*, the ethyldeneamine hydrate.

MISCELLANEOUS.

Royal Institution.—On Thursday next (January 20), Professor Dewar, F.R.S., will begin a course of three lectures at the Royal Institution on "The Halogen Group of Elements."

International Congress of Applied Chemistry.—The Honorary President of the Congress is Professor Dr. A. Bauer, the President is Dr. F. Perger, and the General Secretary is Dr. F. Strohmmer. It is desired to form a committee of English chemists who are likely to be present at the Congress as representatives of England. Dr. F. M. Mrazsek will be happy to give any information about the Congress, including the best and cheapest routes to Vienna, the hotel accommodation, &c., and introductions to the members. He can be consulted personally at 29, Mincing Lane, between the hours of 10 and 5, or by letter.

City and Guilds of London Institute.—The report of the work of the Examination Department for the Session 1896-97 shows a marked increase not only in the number of classes, but also in the number of students and of candidates for examination. The past session has been marked by some extension of the Institute's work in India and the Colonies. Examinations have been held this year in New South Wales and New Zealand, and applications have been received for the holding of examinations in the colonies of Victoria and Queensland. The number of students in attendance during the year shows a very satisfactory increase, being 32,566 as against 29,494 in the previous year: 256 prizes have been awarded this year, comprising 227 money prizes, besides 103 silver and 153 bronze medals. Several changes have been introduced into the programme for the current session, especially with regard to textile subjects. Iron and steel manufacture has also received attention, modifications in the syllabus have been introduced having regard to local conditions of manufacture. Mine surveying, again, is being made more strictly technical; pure mathematics, which have nothing to do with the case, being omitted.

MEETINGS FOR THE WEEK.

TUESDAY, 18th.—Society of Arts, 4.30. "My Recent Journey from the Nile to Souakim," by Frederic Villiers.

— Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

WEDNESDAY, 19th.—Society of Arts, 8. "The Projection of Luminous Objects in Space," by Eric Stuart Bruce, M.A. Microscopical, 8. Annual Address of the President.

— Institution of Mining and Metallurgy, 8. "Notes on the Action of Cyanogen on Gold," by James Park. "Mexican Methods of Mining," by S. B. Newall. "On the Successful Treatment of Tailings by the Direct Filling Process on the Witwatersrand," by F. Cardell Pengelly.

THURSDAY, 20th.—Chemical, 8. Ballot for the Election of Foreign Members. "The Action of Caustic Alkalies on Amides," by J. B. Cohen, Ph.D., and E. Brittain, B.Sc. "The Formation of Monomethyl-aniline from Dimethylaniline," by J. B. Cohen, Ph.D., and H. T. Calvert, B.Sc. "Note on the Aluminium-mercury Couple," by J. B. Cohen, Ph.D., and H. T. Calvert, B.Sc. "Action of Chloroform and Alkaline Hydroxides on the Nitro-benzene Acids," by W. J. Elliott, M.A. "Researches on the Terpenes—II. On the Oxidation of Fenchene," by J. A. Gardner, M.A., and G. B. Cockburn, B.A. "The Preparation of Pure Iodine," by B. Leas, D.Sc., and W. H. Whatmough.

— Royal Institution, 3. "The Halogen Group of Elements," by Prof. Dewar, M.A., LL.D.

— Society of Arts, 4.30. "Recreations of an Indian Official," by the Right Hon. Sir Mountstuart Elphinstone Grant Duff, G.C.S.I., C.I.E., F.R.S. (This meeting will be held in the East Conference Hall of the Imperial Institute, South Kensington).

FRIDAY, 21st.—Physical, 5. "On Electric Signalling without Conducting Wires," by Prof. O. Lodge, F.R.S. A Tesla Oscillator will be exhibited by Prof. S. P. Thompson, F.R.S.

— Royal Institution, 9. "Buds and Stipules," by The Right Hon. Sir John Lubbock, Bart, M.P., D.C.L., LL.D., F.R.S.

SATURDAY, 22nd.—Royal Institution, 3. "Cyprus," by Prof. Patrick Geddes, F.R.S.E.

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CONTENTS.

ARTICLES:—	PAGES
An Improved Superheater for Laboratory Purposes, by J. Arthur Wilson	25
A Note on some further Determinations of the Dielectric Constants of Organic Bodies and Electrolytes at very Low Temperatures, by James Dewar, M.A., LL.D., F.R.S., &c., and J. A. Fleming, M.A., D.Sc., F.R.S., &c.	25
On Cerium, by M. Boudeuard	27
On the Analysis of Silicates, by A. Leclère	27
On the Dissociation Spectra of Melted Salts.—Metalloids: Chlorine, Bromine, and Iodine, by A. de Gramont	28
A Revision of the Atomic Weight of Cobalt, by Theodore Wm. Richards and Gregory Paul Barter	30
Phosphoric Acid Determination — a Gravimetric Method of Estimating Phosphoric Acid as Ammonium Phosphomolybdate, by Thomas S. Gladding	32
London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.	33
NOTICES OF BOOKS.—The Phase Rule	34
CORRESPONDENCE.—Molecules and Liquefaction Heats	35
CHEMICAL NOTICES FROM FOREIGN SOURCES	35
MEETINGS FOR THE WEEK	36

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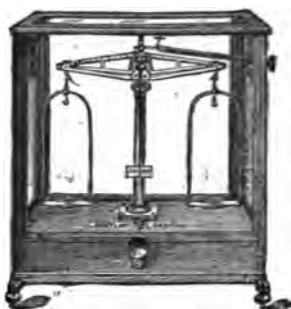
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AN IMPROVED SUPERHEATER FOR LABORATORY PURPOSES.

By J. ARTHUR WILSON.

For laboratory distillation with superheated steam the usual apparatus is a copper coil heated by a Bunsen burner. I have found this plan rather unreliable, as the temperature of the steam is subject to considerable fluctuation. A better plan, which gives very satisfactory results, is to place the coil in a copper cylindrical vessel containing paraffin-wax or similar body. The vessel is heated by a powerful burner, and the temperature equalised by a copper stirrer.

Newchurch, January 7, 1898.

A NOTE ON SOME FURTHER DETERMINATIONS OF THE DIELECTRIC CONSTANTS OF ORGANIC BODIES AND ELECTROLYTES AT VERY LOW TEMPERATURES.*

By JAMES DEWAR, M.A., LL.D., F.R.S., Fullerian Professor of
Chemistry in the Royal Institution,

and
J. A. FLEMING, M.A., D.Sc., F.R.S., Professor of Electrical
Engineering in University College, London.

(Continued from p. 15).

We have, in addition, repeated and extended experiments made with the cone condenser on various electrolytes and dielectrics.

In the first place, we have carefully examined the effect of change of temperature on the dimensions of the cone condenser *per se* to ascertain if the dimensional change produced by cooling it in liquid air could sensibly affect the value of the dielectric constant of an electrolyte forming the dielectric between the cones, apart from the change which temperature produces in the dielectric quality of the dielectric itself.

The gilt cone condenser was accordingly connected with the tuning-fork interrupter as formerly described (see Fleming and Dewar, *Roy. Soc. Proc.*, lxi., 1897, p. 299), and the galvanometer scale deflection when the condenser was charged with 97.2 volts was found to be 3.85 c.m. to the left and 3.88 c.m. to the right,—hence the mean galvanometer deflection was 3.87 c.m. of the scale. This corrected to 100 volts becomes 3.98, and deducting 0.4 c.m. for the capacity of the leads gives 3.58 as the number representing the electrical capacity of the cone condenser as then arranged. A standard condenser belonging to the Davy-Faraday laboratory, and having a capacity of one-thousandth of a microfarad, was then substituted for the cone condenser, and gave right and left deflections of 19.1 and 19.6 c.m. respectively when charged with 97 volts. This corrected for capacity of leads (=0.4 c.m.) and reduced to 100 volts becomes 19.53. Hence, since the electrical capacities are proportional to the galvanometer deflection, the electrical capacity of our cone condenser is—

$$\frac{3.58}{19.53} \times 0.0001$$

of a microfarad, or 0.000183 of a microfarad.

The capacity of the gilt cone condenser was then again

measured with air at 20° C. as dielectric, and the galvanometer deflection observed. The outer cone was then cooled to -185° C. by quickly applying to it a large quantity of liquid air whilst the inner cone remained at about 20° C., and the galvanometer deflection again observed. This deflection was taken again when the inner cone had fallen in temperature to -75°, and finally when both inner and outer cones were at -185° C. The following numbers giving the galvanometer deflections are then proportional to the electrical capacity of the condenser between the cones.

TABLE V.—*Examination of the Effect of Cooling on the Electrical Capacity of the Cone Condenser.*

Galvanometer scale deflection in c.m. or electrical capacity of the cone condenser in arbitrary units, the dielectric being gaseous air.	Remarks.
4.22 c.m. . . .	Inner and outer cones both at 20° C.
4.82 c.m. . . .	Outer cone at -185° C. Inner cone at 20° C., about.
4.58 c.m. . . .	Outer cone at -185° C. Inner cone at -75° C.
4.19 c.m. . . .	Outer and inner cones both at -185° C.

Hence it is clear that mere change of temperature of the metal work of the cone condenser does not affect its electrical capacity by more than 1, or perhaps 2, per cent, and any larger changes in capacity found on cooling must be due to a real change in the dielectric constant of any dielectric substituted for the air between the cones, and not to mere dimensional changes of the condenser itself produced by the cooling.

Another matter to which our attention was directed was the question whether there was any sensible or serious lag in the temperature of the resistance thermometer behind the temperature of the dielectric. Our usual custom had been to immerse the condenser when prepared for use in liquid air, and cool down the whole mass to -185° C., and then raising it out of the liquid air to take temperature and capacity readings as it warmed up. The resistance thermometer was placed in the inner cone in a thin test-tube, and fixed in with fusible metal in the inner cone. We therefore tried one experiment with pure glycerin as dielectric, in which the electrical measurements were made as the condenser was slowly cooled, instead of being made as it slowly heated up. The process of cooling from -38° pt. to -201° pt. was allowed to occupy one hour and forty minutes. The values thus found for the dielectric constant for glycerin for this range of temperature were practically in agreement with those found when the condenser capacity was measured as it warmed up instead of cooled down.

TABLE VI.

I. *Dielectric Constant of Pure Glycerin.*

Corrected galvanometer deflection when the condenser had
air as dielectric = 3.92 c.m. for 100 volts.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
- 38°	2.85	50.5	Condenser charged with 1.434 volts. Time = 3.5 p.m.
- 42°	2.97	52.8	
- 46°	3.00	53.3	
- 55°	3.17	56.5	
- 64°	3.03	53.8	Time = 4.15 p.m.
- 98°	2.70	3.95	Condenser charged with 17.0 volts.
- 119°	2.20	3.19	Time = 4.30 p.m.
- 201°	11.05	2.82	Condenser charged with 92.4 volts. Time = 4.45 p.m.

* A Paper read before the Royal Society, December 9th, 1897.

The above values of the dielectric constant of glycerin are in very fair agreement with the values obtained by us during rising temperature (*Roy. Soc. Proc.*, lxi., p. 324).

We have also extended our former observations made with this cone condenser and the galvanometric method to certain other frozen electrolytes, and measured their dielectric constants at low temperatures.

The results and substances are as follows :—

II. Dielectric Constant of Dry Concentrated Sulphuric Acid (H_2SO_4).

The corrected galvanometer deflection with air as dielectric was 3.92 c.m. for 100 volts. The switch frequency was 120.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
-200.9	14.6	3.86	Condenser charged with 94.2 volts.
-186.0	14.8	3.90	Condenser charged with 94.2 volts.
-181.8	2.5	3.82	Condenser charged with 16.2 volts.
-150.7	2.7	4.13	Condenser charged with 16.2 volts.
-129.8	2.95	4.33	Condenser charged with 16.9 volts.
-110.0	6.0	7.25	Condenser charged with 17.0 volts.

III. Dielectric Constant of Dry Concentrated Nitric Acid (NO_3H).

Corrected galvanometer deflection with air as dielectric = 3.90 c.m. for 100 volts.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
-201.7	9.05	2.36	Condenser charged with 94.2 volts.
-182.7	9.27	2.46	
-165.8	9.25	2.42	
-118.2	9.40	2.45	
-129.7	10.00	2.62	

Before carrying out these experiments with the concentrated nitric and sulphuric acids the condenser had been carefully re-gilt, and a glass steady-pin substituted for the ebonite one.

IV. Dielectric Constant of Sodium Fluoride (NaF). (10 per cent solution).

Corrected galvanometer deflection with air as dielectric = 4.04 c.m. for 100 volts.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
-199.3	9.3	2.33	Condenser charged with 94.2 volts.
-174.2	9.3	2.35	
-149.2	10.35	2.55	
-135.0	14.35	3.67	
-125.0	19.6	5.05	
-115.7	4.4	7.07	Condenser charged with 15.2 volts.

The electrical resistance of the condenser at -200 $^{\circ}\text{F}$. with frozen sodic fluoride as dielectric was 2000 megohms.

V. Dielectric Constant of Hydrosodic Fluoride (NaHF). (10 per cent solution).

Corrected galvanometer deflection with air as dielectric = 3.42 c.m. for 100 volts.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
-201.8	8.87	2.28	
-186.0	8.87	2.25	
-169.0	8.95	2.30	
-178.8	9.0	2.31	
-148.8	9.7	2.49	
-142.2	10.9	2.79	
-131.5	15.4	4.02	

VI. Dielectric Constant of Sodium Peroxide (Na_2O_2). (5 per cent solution).

Corrected galvanometer deflection with air as dielectric = 4.41 c.m. for 100 volts.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
-198.0	4.5	71	Condenser charged 1.434 volts.
-184.5	5.5	87	

VII. Dielectric Constant of Solution of Hydroxyl (H_2O_2). (20 per cent solution by volume).

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
-203.5	10.8	2.38	Condenser charged with 99.2 volts.

Some experiments were then made by electrolyzing freely certain electrolytes and freezing them with liquid air in the act of electrolysis. The dielectric constants were then subsequently determined in the frozen state.

VIII. Dielectric Constants of Frozen Electrolytes, Electrolysed freely in the act of Freezing.

Corrected galvanometer deflection with air as dielectric = 3.42 c.m. for 100 volts.

Temperature in platinum degrees.	Mean galvanometer deflection in centimetres.	Dielectric constant.	Observations.
(a) 5 per cent aqueous solution of potassium hydrate electrolysed with 0.2 ampère and 8 volts. Evolved gas = 5.1 c.c.			
-200.0	3.85	71.4	Condenser charged with 1.434 volts. Electrical resistance of condenser 5000 megohms when frozen.
(b) Water electrolysed with 1.0 ampère.			
-198.2	8.70	2.47	Condenser charged with 98 volts. Electrical resistance of condenser 5000 megohms when frozen.
(c) Water electrolysed with 2.1 ampères.			
-198.0	8.65	2.42	Condenser charged with 98.9 volts.

It is evident that the action of electrolysis prior to and during freezing has no sensible effect on the subsequently measured dielectric constant, even though the surfaces of the cone condenser are strongly polarised in the act of freezing the liquid dielectric.

We have then paid some attention to the possible cause of the high dielectric values of some substances at very

low temperatures. It is clear from the above-described experiments with the Nernst bridge that for organic bodies such as ethylic alcohol, amyl alcohol, ethylic ether, and glycerin, we obtain practically the same dielectric values at the low temperature, both when they are measured by the galvanometric method with a switch frequency of 120 and when measured by Nernst's method with a frequency of 350, or about three times as great.

On the other hand, for certain other bodies, such as the frozen dilute hydrates of potassium and rubidium, and the oxide of copper suspended in ice, the dielectric value at the low temperatures is much diminished by increasing the frequency.

(To be continued).

ON CERIUM.

By M. BOUDOUARD.

In a note presented to the Académie (*Comptes Rendus*, vol. cxxv., p. 950) MM. Wyruboff and Verneuil criticised the results I obtained with cerium (*Comptes Rendus*, vol. cxxv., p. 772).

I. While remarking on the difference in the origin of the oxides which were used by MM. Wyruboff and Verneuil and myself, I will show, first of all, that my analytical results did not present between themselves any greater differences than those of these gentlemen.

MM. Wyruboff and Verneuil pointed out as an isolated fact that in the fractionations of the sulphate the mother-liquors gave figures sometimes higher and sometimes lower than those furnished by the crystalline deposits. In their own determination we observe absolutely the same facts either in one way or the other.

For example, MM. Wyruboff and Verneuil found:—

I.	{ 1st crystallisation 92.49 } $d=0.27$; $\epsilon=\frac{0.27}{92.5}=0.0029$
	{ 3rd " 92.76 }
II.	{ 1st " 92.56 } $d=0.74$; $\epsilon=\frac{0.74}{93}=0.0079$
	{ 3rd " 93.3 }

For my part, I obtained—

I.	{ 1st crystallisation 136.5 } $d=0.9$; $\epsilon=\frac{0.9}{137}=0.0065$
	{ Mother-liquors 137.4 }
II.	{ 1st crystallisation 137.15 } $d=0.45$; $\epsilon=\frac{0.45}{137}=0.0032$
	{ Mother-liquors 137.6 }
III.	{ 1st crystallisation 137.85 } $d=2.05$; $\epsilon=\frac{2.05}{139}=0.0144$
	{ Mother-liquors 139.9 }

Thus, MM. Wyruboff and Verneuil qualify as isolated a fact given in my note, analogous results being also given in their memoir. I would remark that the error in both cases is equally great.

2. As to the difficultly acceptable observation that a cerium with an atomic weight of 137.85 has given two parts, of atomic weights of 140.7 and 138.5, that was a mistake on my part in writing the article only; there was nothing in common between the two series of determinations, and it should read—"Having made crystallisations of a sulphate prepared from a basic acetate," instead of "having made crystallisations of this same sulphate."

3. As to the action of peroxide of hydrogen on the acetate of cerium, as I always operated at a warm temperature I obtained a partial precipitation; that is, in fact, what I intended to do—it was not an error of observation. As to the purity of the oxide of cerium with which I worked, I believe it to be quite beyond criticism. MM. Wyruboff and Verneuil insist that the criterion of purity of oxide of cerium is that it should be of a white colour; the oxide of cerium which I have obtained is white, and I took a great deal of trouble to free the cerium, not only from thorium, but from the other earths of the cerium and yttrium groups. I prepared the oxide

of cerium by Debray's method, and it was the salts of this purified oxide that I fractionated.—*Comptes Rendus*, vol. cxxv., No. 25.

ON THE ANALYSIS OF SILICATES.

By A. LECLÈRE.

THE principal difficulty in the analysis of silicates is in the passage of the silica through the gelatinous state. We well know that the evaporation to dryness, to which we ultimately have recourse, tends to the partial entanglement of the bases in the residue.

The analogies which exist between silicon, titanium, and tin would lead us to suppose that it is possible to obtain silica directly in the insoluble state by attacking a silicate, which would not become hydrated during its decomposition, with sufficiently concentrated nitric acid. We have proved that this result can be obtained by previously melting natural silicates with oxide of lead: this base, at a moderate temperature, forms very fusible compounds with all the elements found as silicates, and it retains the alkalis in a well-marked and characteristic manner.

It has been pointed out that the presence of oxide of lead is injurious to platinum vessels; but we have proved that platinum is in no way injured if we use pure oxide of lead, prepared in the manner which we shall presently describe, and also conduct the fusion in a muffle furnace in which any contact between the platinum and the gas flame is absolutely guarded against.

Porphyrised silicate is mixed with oxide of lead; a convenient quantity of oxide for a refractory clay is about three times the weight of the mineral, but the presence of alkalis enables us to reduce this proportion. We used a 40 m.m. platinum crucible weighing 4 grms., and provided with a cover. The fusion takes about half an hour in a muffle at a reddish orange heat, but if the silicate has not been finely powdered it is as well to prolong the fusion and add a little more oxide. By this means we obtain a liquid enamel which is easily detached from the platinum if we cool the underneath part quickly, taking care to keep the cover on, so as to avoid any loss by decrepitation.

This enamel is completely decomposed by a mixture, of not less than ten times its weight, of equal parts of ordinary nitric acid and fuming nitric acid. The attack should be made either in the cold or at about 40°, and if the enamel is first of all powdered it lasts about an hour; if, on the contrary, the pieces are comparatively large, the operation takes a longer time, even as long as one or two days. The attack is always complete, and leaves a residue composed of nitrate of lead and of completely insoluble hydrated silica.

Nothing now remains but to dilute with boiling water, which entirely dissolves the lead, and to collect the silica on a filter. The termination of the washing can be decided by means of a glass rod moistened with sulphide of ammonium, and the silica can be weighed after ignition at a high temperature.

The insoluble hydrate obtained by this method retains about 20 per cent of moisture when dried at 100°. If we operate on pure silica, and prepare the enamel in sufficiently thin sheets, so that there is no swelling under the action of the acid, we can obtain plates which show all the characteristic colours of the opal, after the solution of the nitrate of lead and the subsequent drying.

The acid liquor containing the nitrates is concentrated to get rid of the excess of nitric acid, and alcohol is then added. A quantity of hydrochloric acid, a little more than is necessary to precipitate the lead, is then added, and the lead separates immediately. The alcoholic liquor leaves on evaporation a residue, on which the estimation of the bases can be proceeded with by the well-known methods of Sainte-Claire Deville and M. Schloesing.

The oxide of lead should be prepared according to the following method:—Into a 15 per cent solution of commercial nitrate of lead we pour a saturated solution of oxalic acid, containing also about 3 per cent of nitric acid. A very dense precipitate of oxalo-nitrate of lead is immediately formed, the impurities remaining in the acid solution. The precipitate is separated, dried in the oven, and calcined at a little below red heat in a porcelain crucible. About one-quarter of this is taken, moistened with nitric acid, and then ground up with the remainder. By repeating the calcination we obtain a finely powdered minium, which constitutes the best form of lead oxide for the decomposition of silicates. — *Comptes Rendus*, vol. cxxv., No. 22.

ON THE DISSOCIATION SPECTRA OF MELTED SALTS.

METALLOIDS: CHLORINE, BROMINE, AND IODINE.

By A. DE GRAMONT.

Chlorine.

THE spectrum of free chlorine has been studied and measured by the late M. Salet in tubes at the ordinary pressure, excited by means of a spark from either an influence machine or a Ruhmkorff coil without a condenser. This question has also been studied by M. Thalén, and afterwards by M. Hasselberg under similar conditions, but with greater accuracy.

Having obtained, with the apparatus which I have already described in a previous paper, very excellent spectra of the lines of the metalloids in free air, without having recourse to Plücker or Salet tubes, but by the dissociation of melted salts under the influence of a condenser spark, I can now give the spectrum of chlorine produced by the principal alkaline chlorides, in which the small number of metallic lines renders the study much more simple. I still name the principal groups of lines by the Greek letters and the numbers used by M. Salet.

For comparing these figures with those of previous observers, I must refer my readers to a more extended memoir which I published in the *Annales de Chimie et de Physique*, Series 7, vol. x., Feb., 1897.

The following figures have been reduced to the normal spectrum of Rowland by means of a correction of +0.1.

Cl a	610.6	Doubtful.	
Cl β	1. 545.7	Very well marked.	
	2. 544.4	Fairly strong.	
	3. 542.4	" "	
	4. 539.3	" "	
Cl γ	532.6	Very faint; of doubtful origin.	
	522.1	Strong.	First principal group.
	521.7	" "	
	510.4	Fairly well seen.	
Cl δ	510.0	" "	
	2. 507.8	Easily seen.	
	499.5	Fairly well seen.	
	497.4	Rather weak.	
Cl ε	492.5	Weak.	
	491.7	Easily seen.	
	2. 490.4	Fairly strong.	
	3. 489.8	" "	
Cl ζ	482.1	Strong; bright.	Second principal group.
	481.1	" "	
	479.5	" "	
	478.2	Well marked.	
Cl η	476.9	" "	
	471.4	Faint.	
	457.3	Diffuse; faint.	

All the chlorides tried have given the lines of the spectrum of chlorine with the greatest facility, with the

exception of those, of course, which were effaced by being in the immediate neighbourhood of, or in apparent coincidence with, the intense metallic lines, but these lines have been found by using different metals. The measurements were made with NaCl, LiCl, KCl, RbCl, CdCl₂, and ZnCl₂.

The Group Cl β is characteristic, and easily seen with even a small quantity of chlorine in the substance under examination. Cl γ—which is formed of two very bright strong lines, but which cannot be separated in an apparatus with only one prism—appears to be the most persistent both in duration and sensibility, and with Cl ζ the most characteristic. Cl δ, which is easily seen, is formed of three series; the first two of which appear as one with only one prism, are less visible than the third. The two most refrangible single lines of Cl ε are fairly bright. Cl ζ comes immediately after Cl γ in importance and duration, and is, in fact, of almost equal rank. This group is essentially characteristic from the analytical point of view, and the most easy to recognise on account of the position of its three very strong and bright blue lines. Br μ, which occupies nearly the same position, consists of two lines only.

As a whole, the spectrum of chlorine is more beautiful, and the lines brighter and sharper, in melted chlorides with the condenser spark than in tubes with free chlorine, when they appear to be so diffuse that those nearest to each other cannot be separated.

As an example of the sensitiveness of the spectral reaction of chlorine by this method, we may mention this fact:—A bead of 0.02 grm. of Na₂CO₃, containing 0.0005 of chlorine added in the form of NaCl, gave all the lines of chlorine; with only 0.0001 grm. of chlorine the groups Cl γ and Cl ζ were still visible. Hydrogen prepared from hydrochloric acid and zinc will, when the flow of gas through the wash-bottles is too rapid, carry a certain amount of hydrochloric acid with it, the traces of which can be detected by the appearance of Cl γ.

Bromine.

Since the memoir published by Salet, in 1873, I am not aware that the spark spectrum of bromine has been the subject of any further research. Richer in lines than that of chlorine, it is also as bright and as easily observed in melted salts dissociated by the condenser spark.

I here give the wave-lengths measured with a direct-vision spectrocope with two prisms, and corrected to the normal spectrum of Rowland. The arrangement of the apparatus was the same as in the previously described experiments. (See Table, next column).

The Greek letters and the numbers used are the same as those of M. Salet. Br β, easily seen, is given by M. Salet as a triple line; he, however, only used a single prism. On the contrary, I, with my spectrocope with two fairly dispersive prisms, with a very narrow slit, and a very steady support, have never been able to see more than one line in this position; it is very sharp in melted bromides, but vague in a tube of free bromine. Again, Br γ, Br δ, and Br ε, described as double by M. Salet, have not been noticed as double by me under the conditions just described, although the very greatest care was used. The brilliancy of Br γ is slightly diminished by the almost inevitable presence of Na a. Br δ appears to be very much weakened in the condenser spark, and is difficult to see. The group Br ζ is very characteristic, and has two of its lines (ζ₁ and ζ₂) much more persistent than the others. Br η and Br θ appear to be much brighter than they were described by M. Salet. The two characteristic lines Br μ are very distinctive of bromine, although they are very close neighbours of some of the chlorine lines, which actually has one in between the two. Br ν, which is very strong, appears to be the most persistent in the case of only a very small percentage of bromine being present in the salt under examination. Br π, easy to see, terminates the visible spectrum of bromine under the conditions in which

Br α	632.5	Well marked.
Br β	614.7	Easily seen.
	587.1	Faint; doubtful.
Br γ	583.05	Well marked.
Br δ	572.0	Very faint.
Br ϵ	559.05	Well marked.
	551.1	Easily seen.
	549.85	" "
	549.2	" "
	544.8	Faint.
	543.6	Fairly well seen.
	542.5	Easily seen.
Br ζ	1. 533.2	Very strong.
	2. 530.5	Strong.
	3. 523.75	Very strong.
	4. 518.35	" "
	5. 516.5	Well marked.
	510.75	Fairly strong, but variable.
Br η	505.5	Well marked.
Br θ	493.0	Fairly strong.
Br μ	1. 481.7	Very strong.
	2. 478.7	" "
	476.8	Easily seen.
	474.4	" "
	472.1	" "
Br ν	470.45	Very strong.
	469.25	Fairly well seen.
	467.75	Fairly strong.
	462.3	Easily seen.
Br π	436.55	" "

I worked. The most characteristic groups of this metal-iodine in melted salts are therefore:—

Of first rank Br ν , Br μ .
Of second rank Br ζ .

The measurements were made on NaBr, KBr, CaBr₂, ZnBr₂. The sensitiveness of the spectral reaction of bromine seems to be equal to that of chlorine, and has been similarly used in analysis.

Iodine.

Like bromine, iodine has not been studied since the research of M. Salet on the spark spectrum with an ordinary monoprismatic spectroscope. I here give the spectrum of this body obtained by the dissociation of NaI, KI, and CdI₂, with the condenser spark, and my usual apparatus, and spectroscope.

The wave-lengths have been corrected for the normal spectrum of Rowland:—

	625.8	Fairly well marked
	620.7	" "
I α	612.7	Very well marked.
I β	608.6	Fairly visible.
	607.7	Fairly strong.
I γ	595.1	Strong.
	578.9	Fairly visible.
I δ	1. 577.55	Well marked.
	2. 576.1	Fairly well marked.
	3. 573.85	Well marked.
	4. 571.0	" "
	5. 569.2	Fairly well marked.
I ϵ	567.85	Well marked.
	562.7	" "
	561.4	Very faint.
	560.2	Faint.
	559.6	" "
	555.4	Very faint.
	552.5	" "
	550.8	Faint.
I ζ	1. 549.9	Strong.
	2. 549.6	Well seen.
	3. 546.6	Very strong.
	4. 543.8	Strong.

	537.0	Fairly strong.
I η	1. 534.4	Very strong.
	2. 533.75	" "
	526.8	Well seen.
I θ	526.45	Fairly well seen.
	524.55	Well marked.
	521.65	" "
	517.65	Faint.
I μ	516.25	Very strong.
	510.7	Well marked.
I ν	506.6	Fairly well seen.
I ξ	1. 486.5	Well seen; diffuse.
	2. 485.05	" "
	480.55	Faint.
	476.6	" "
	473.25	" "
I σ	467.75	Well marked.
	466.8	" "
I π	464.25	" "
	463.2	" "
	462.3	" "
I ρ	445.4	Fairly well seen; diffuse.
	445.0	" "
	444.0	Rather faint; diffuse.
	441.15	Faint; diffuse.
I ϵ	422.5	Well seen.

As is at once seen, this spectrum is richer in lines than those of both chlorine and bromine, particularly in the orange red. The principal lines in this region, I α , I β , I γ , are easily seen, even with a low proportion of iodide in the salt under examination—a fact which does not apply to the two red lines in the bromine spectrum. I δ , formed of seven lines, is not particularly bright. I ϵ is, on the contrary, very bright, as is also the group I ζ , and especially the line ζ_2 (546.6).

Chlorine has a group (Cl β) almost corresponding in appearance and position with I ζ , but it is not so bright, and the lines are not so wide apart as in the latter case. I give 549.6 with all reserve, because of the air line 549.66 (Néovius), although the air spectrum was not apparent at the time of the observation. I η , consisting of two brilliant lines, and the strong line I μ , are the most remarkable in the spectrum of iodine, both by reason of their intensity and their sensitiveness in detecting minimal quantities of iodine. (521.65) almost coincides with Cl γ (521.70), the most sensitive group in the chlorine spectrum; but experience has shown that it is really due to iodine, where I have always found it of constant intensity, and unaccompanied by the other component (522.10) of Cl ζ . I θ , I ν , I σ , I ρ , and I ω are well seen and of medium brilliancy, superior to the diffuse lines of the group I γ , which are often difficult to separate.

The most sensitive and characteristic groups or lines of iodine in melted salts are therefore:—

Of first rank I η , I μ , I ζ .
Of second rank I ζ (as a whole),
I α , I β , I γ .

The spectral sensitiveness of iodine, tested by the method already used for chlorine, seems to be about the same.—*Bull. Soc. Chimie*, Series 3, vol. xvii-xviii, No. 20.

A Coloured Reaction of Ordinary Aldehyd.—Louis Simon.—The property of producing a beautiful red colouration with nitro-prussiate of sodium and potash, either with or without the subsequent addition of acetic acid, is shared by ordinary ethylic aldehyd with a large number of aldehydic and ketonic bodies; but, on the other hand, the following reaction, viz., adding a few drops of aqueous methylamine and a few drops of dilute nitroprussiate to a weak solution of ordinary ethylic aldehyd, gives a beautiful blue colour which is quite characteristic.—*Comptes Rendus*, cxv., No. 25.

A REVISION OF THE ATOMIC WEIGHT OF COBALT.*

FIRST PAPER.—THE ANALYSIS OF COBALTOUS BROMIDE.

By THEODORE WILLIAM RICHARDS

and

GREGORY PAUL BAXTER.

(Concluded from p. 23).

The Method of Analysis.

THE most insidious of all impurities in accurate quantitative work is water. Its insidiousness is due to the difficulty of detecting it in small amounts, as well as to its invariable occurrence in the atmosphere and its common use as a solvent and medium for crystallisation. Our first analytical problem, in this case just as in the case of nickel, was to weigh the salt to be analysed in such a fashion as to exclude this ever watchful enemy. The fact that cobaltous bromide is less easily decomposed at high temperatures than is nickelous bromide, tended to make our present problem the easier of the two; but the much greater hygroscopicity of the cobalt salt had the opposite tendency. The apparatus so useful in the cases of magnesium (Richards and Parker, *Proc. Amer. Acad.*, xxii., 59), zinc, and nickel (Richards and Cushman, *Proc. Amer. Acad.*, xxxiii., p. 95; *CHEM. NEWS*, lxxvi., p. 284), proved to be equally serviceable here; cobaltous bromide, after being ignited in a stream of mixed nitrogen and hydrobromic acid, cooled in dry nitrogen, and bottled automatically in dry air, may be weighed with perfect certainty. The bottling apparatus has been described so often that further details are unnecessary here; but the apparatus for supplying the desired gases has not yet been explained in all the complication of its present form.

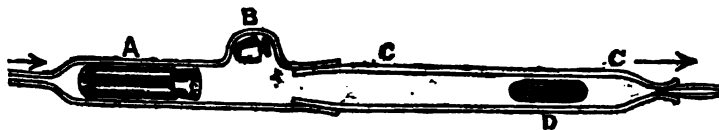


FIG. 2.—BOTTLING APPARATUS, HORIZONTAL SECTION.

A, Weighing bottle. B, Stopper of bottle. C, Hard glass tube. D, Platinum boat containing cobaltous bromide.

In the first place, nitrogen was prepared by passing air through strong ammonia water, and then over red-hot copper. The nitrogen, after passing through several flasks of sulphuric acid to remove the excess of ammonia, was conducted into column 1 (see Fig. 3), containing beads soaked with silver nitrate, in order to free the gas from the possible admixture of sulphur compounds taken from the rubber connections which were used in the apparatus for making nitrogen. In the columns numbered 2 were beads drenched with strong sulphuric acid, which thoroughly dried the nitrogen. With stopcock 12 open, the nitrogen passed over phosphoric anhydride in tube 11 directly into the bottling tube. With stopcock 12 closed and 4 open, the nitrogen of necessity bubbled through bromine in the small flask 5; beyond this flask, if stopcock 9 were open, the gases passed directly over phosphoric anhydride in 10 to the cobalt bromide; if stopcock 9 were closed, the nitrogen and bromine together passed into the flask 6, containing hydrobromic acid and red phosphorus, thence into the U-tube 7, containing beads moistened with hydrobromic acid and red phosphorus, thence over anhydrous calcic bromide in the tube 8 to remove the water taken from the hydrobromic acid, and finally over phosphoric anhydride in 10 to the bromide of cobalt. Dry air was prepared by passing a current of air through several columns of beads (O P in the figure) moist-

ened with sulphuric acid, and then into the apparatus through the stopcock 14. The pressure of the sulphuric acid in the bulb 3 prevented any air from backing up into the sulphuric acid columns delivering nitrogen. In order to prevent the slightest admixture of hydrobromic acid with the dry air used to wash out the apparatus at the conclusion of the operation, a slight excess of pressure was always maintained within the apparatus. This excess of pressure was so regulated as to cause a part of the pure air to go backwards through tube 10 (stopcocks 4 and 9 being closed), and to bubble through the sulphuric acid kept in the vessel labelled 13. The tube and vessel served as a safety-valve and pressure gauge throughout the whole operation. The ground joints were kept tight by means of syrupy phosphoric acid.

To the right hand end of the complex apparatus here described was fused the automatic bottling apparatus, as indicated in Fig. 3. By means of this arrangement the cobaltous bromide could be thoroughly dried, and enclosed in a suitable bottle for weighing without a moment's exposure to the moist air of the room. In one case, where a platinum boat containing several grms. of the salt was re-heated and re-bottled after weighing, the loss in weight was found to be only 0.11 milligram., an amount not greater than the probable loss by sublimation at the high temperature (400°) employed in the ignition. Hence the apparatus evidently answers its purpose well.

The preliminary analyses were so much easier to make than those described in the preceding paper—because of the ready solubility of the salt used, and its invariable freedom from the oxide whose presence had caused trouble at first in the work upon nickel—that only three were deemed necessary.

The method of procedure may be easily gathered from previous descriptions. The bromides of cobalt and silver

were each weighed, as in the case of the nickel, and from the assumed molecular weight of argentic bromide 187.885 that of cobaltous bromide was calculated.

From the value for the atomic weight of cobalt obtained from this preliminary series, the approximate proportion of silver necessary for the complete decomposition of a given weight of cobaltous bromide was calculated; and in the succeeding analyses the proper amount of silver was weighed out in each case. After the silver had been dissolved with precautions to prevent loss during the process, and after the solutions of cobaltous bromide and argentic nitrate had been much diluted, the precipitation was effected in a roomy, glass-stoppered Erlenmeyer flask, and the mixture was very thoroughly shaken and allowed to stand. In three experiments, Nos. 4, 6, and 7, the precipitation was completed by titrating backward and forward with centinormal argentic nitrate and hydrobromic acid solution, until the precise point was found (*Proc. Amer. Acad.*, xxviii., 24; xxx., 384); while in Nos. 8, 9, 11, 12, and 13 the nephelometer was used to determine the end-point (*Ibid.*, xxx., 385). Of course, the work was done in yellow light or in darkness, and great care was taken to prevent the loss of any of the precipitate. The results of this determination of the amount of silver required to precipitate the bromine in cobaltous bromide are given in the table headed, "Third Series," below. The "Second Series" of results was obtained by weighing the argentic bromide precipitated in the manner just described. Before filtering, a slight excess of argentic

* Contribution from the Chemical Laboratory of Harvard College. From the *Proceedings of the American Academy of Arts and Sciences*, vol. xxxiii., No. 7.

The Atomic Weight of Cobalt.

O = 16.000; Ag = 107.93.

FIRST SERIES (PRELIMINARY).—2AgBr : CoBr₂.

Number of analysis.	Sample of CoBr ₂ .	Weight of cobaltous bromide in vacuum.	Weight of argentic bromide in vacuum.	Atomic weight of cobalt.
1.	I.	2.25295	3.86818	58.950
2.	I.	2.88763	4.95732	58.975
3.	I.	1.88806	3.24056	59.026
Average				58.984

nitrate was added, and after long agitation the precipitate was collected as usual upon the Gooch crucible. The shreds of displaced asbestos were collected and weighed, and from the total was subtracted the small amount (if any) of argentic bromide resulting from hydrobromic acid added in titration. One experiment (No. 5), in which the cobaltous bromide contained a few visible chips of porcelain from the tube used in subliming, was rejected; and in one (No. 10) the weight of the silver was not determined.

THIRD SERIES.—2Ag : CoBr₂.

Number of analysis.	Sample of CoBr ₂ .	Weight of cobaltous bromide in vacuum.	Weight of silver in vacuum.	Atomic weight of cobalt.
4.	I.	1.33564	1.31702	59.002
6.	I.	2.58129	2.54585	58.955
7.	I.	2.84382	2.80449	58.977
8.	I.	1.83722	1.81170	58.991
9.	I.	2.68584	2.64879	58.969
11.	II.	2.88914	2.84891	58.998
12.	III.	2.32840	2.29593	59.003
13.	IV.	1.91703	1.89033	58.999
Average				58.987

Average of Series II. and III. . . 58.991

which were the most carefully made, the ratio becomes 57.446 : 100.000, while Stas's result is 57.445. The value of this comparison has been pointed out in the previous paper on nickel.

A still more important consideration is the identity of

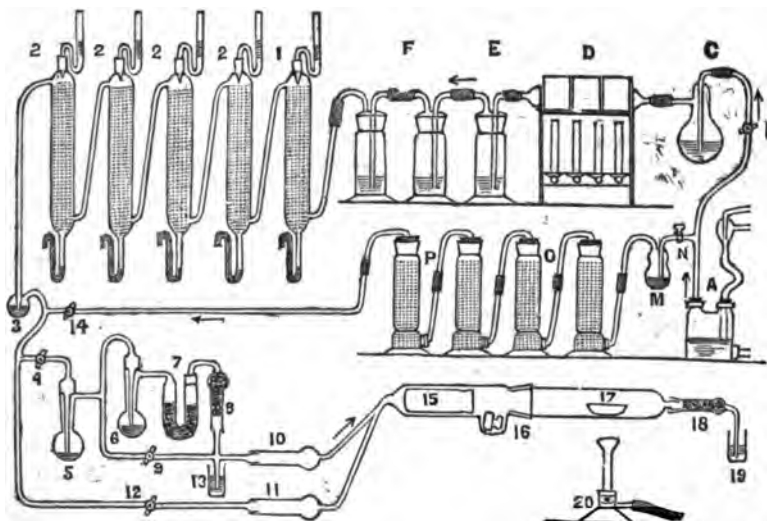


FIG. 3.—APPARATUS FOR IGNITING COBALTOUS BROMIDE IN ANY DESIRED MIXTURE OF GASES. The use of rubber was confined to the first part of this train, where it could do no harm (A B C D E F and A N O P).

As usual in cases of this sort, we may obtain a useful check upon the accuracy of these results by comparing the amount of silver required with the amount of argentic bromide formed. Thus, in all these experiments taken together 18.16302 grms. of silver yielded 31.61642 grms. of argentic bromide—a ratio of 57.448 to 100.000. If we take only the last five experiments (8, 9, 11, 12, and 13),

SECOND SERIES.—2AgBr : CoBr₂.

Number of analysis.	Sample of CoBr ₂ .	Weight of cobaltous bromide in vacuum.	Weight of argentic bromide in vacuum.	Atomic weight of cobalt.
4.	I.	1.33564	2.29296	58.975
6.	I.	2.58129	4.43095	58.998
7.	I.	2.84382	4.88135	59.009
8.	I.	1.83722	3.15368	59.000
9.	I.	2.68584	4.61046	58.996
10.	II.	3.18990	5.47607	58.982
11.	II.	2.88914	4.95943	58.997
12.	III.	2.32840	3.99706	58.987
13.	IV.	1.91703	3.29053	59.010
Average				58.995

the results obtained from the various samples. The average of all the results of each of the four preparations is given below—the first average comprehending ten results, the second three, and the third and fourth each two results :—

Sample I.	58.987
Sample II.	58.992
Sample III.	58.995
Sample IV.	59.004

In this case, as in the case of nickel, a slight rise in the atomic weight is to be observed with the increasing purity of the materials. Although larger here than before, the rise cannot be considered greater than the probable experimental error—especially since one or two of the individual results obtained from Sample I. were slightly greater than any obtained from Sample IV. We must therefore conclude that, if "gnomium" exists, it must have an atomic weight about equal to that of nickel and cobalt, and hence that the wide variations to be observed in the results of other experimenters cannot be considered a valid argument in favour of the late Professor Küss's doubtful discovery.

According to the present investigation then, the atomic weight of cobalt seems to be very close to 59. Upon

comparing this result with the earlier ones of other experimenters, we see that it is as much in accord with their general verdict as almost any other would be. In other words, the values are so various in magnitude that no satisfactory conclusion can be drawn from them. It is worth noticing that one of the results obtained by Thiele, under Professor Hempel's direction, agrees exactly with ours, however.

Our work thus has this outcome at its present stage:—

If Oxygen = 16.000, Cobalt = 58.99.

If Oxygen = 15.88, Cobalt = 58.55.

PHOSPHORIC ACID DETERMINATION.*

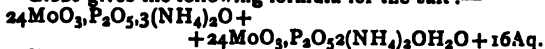
A GRAVIMETRIC METHOD OF ESTIMATING PHOSPHORIC ACID AS AMMONIUM PHOSPHOMOLYBDATE.

By THOMAS S. GLADDING.

THE estimation of phosphoric acid by weighing the yellow precipitate of ammonium phosphomolybdate has often been attempted, but, except in iron analysis, where the amount of phosphorus is very small, such a method has never yet been successful. The reason of such failure is evident when we consider the analyses that have been made of the yellow precipitate. A few only need be presented.

	Rammels- berg.	Struve and Svanberg.	Sonnen- schein.	Gibbs.
H ₂ O	5.77	9.49	11.23	3.94
NH ₄ OH	3.25	9.49	11.23	3.35
P ₂ O ₅	3.90	3.63	3.03	3.66
MoO ₃	86.45	86.88	86.87	89.05

Gibbs gives the following formula for the salt:—



He prepared the salt by mixing solutions of ammonium molybdate (7 parts of water to 3 of the salt) and phosphate, adding nitric acid in excess to the solution, and boiling. Such a method would give more or less occluded molybdic oxide. His analyses gave 3.70 and 3.83 per cent of phosphorus pentoxide against the theoretical percentage of 3.66. Such results were sufficiently accurate for his purposes, but would discourage any suggestion of using the yellow salt as the basis of a gravimetric method. His formula is, however, without any doubt, the correct one, with the exception of the water of crystallisation.

My own analysis of the salt precipitated in the manner described later on, and dried at a temperature of 105° C. to a constant weight, gives me the following composition:—

	Theoretical.	By analysis.
48MoO ₃	91.38	91.36
2P ₂ O ₅	3.76	3.76
10NH ₃	2.25	2.31
11H ₂ O	2.61	2.57

For ammonia, 1.015 grms. gave 0.0234 gm. NH₃ = 2.30 per cent.

For ammonia, 20.193 grms. gave 0.4690 gm. NH₃ = 2.32 per cent.

For moisture, 7.25 grms. gave ——— = 2.53 per cent.

For moisture, 10.30 grms. gave ——— = 2.61 per cent.

The water was determined by estimating total hydrogen by combustion with copper oxide.

For phosphoric acid, repeated analyses and syntheses gave almost exactly 3.76 per cent.

The molybdic acid was found by difference.

The fact that drying at 105° C. expels all the water except eleven molecules, and that the yellow salt when dried over sulphuric acid in a close desiccator comes to the same weight as when dried at 105° C. would indicate

that this salt contains no other water of crystallisation. Any excess of water is apparently hygroscopic water and not water of crystallisation. I therefore find the following as the correct formula for the crystallised yellow salt:—



The following method of procedure has given me a precipitate of a very uniform composition, and would seem to afford the simplest and easiest method yet presented for estimating phosphoric acid.

To the solution of phosphoric acid (25 c.c. to 50 c.c. in bulk) are added 25 c.c. of strong ammonia, 0.900 sp. gr.; nitric acid, 1.42 sp. gr., is now added to acidity. The beaker containing the solution is placed in a water-bath maintained at a constant temperature of 50° C. The ordinary five per cent acid molybdate solution is now added from a burette at the rate of about 3 drops per second, with constant stirring (50 c.c. may be added in five minutes). When the molybdate solution to an excess of about 10 c.c. has been added, the beaker is allowed to remain for ten minutes in the bath. The contents are then filtered through a weighed filtered paper.

The filtrate, without the washings, and after the addition of 5 c.c. molybdate solution, is replaced in the bath for ten minutes. The liquid should remain clear, or at most show only a faint opalescence.

For washing the precipitate, a wash water of dilute nitric acid 100:1 is employed. Three generous washings by decantation and three washings on the paper, followed by one final washing with distilled water are sufficient. The paper and contents are now drained for a few minutes on some waste filter or blotting paper, and then dried to a constant weight at a temperature of 105° C.

In this method the formation of a pure granular precipitate of uniform composition and free from occluded salts is secured by the gradual addition, drop by drop, of the molybdate solution with constant stirring. The completeness of precipitation of the phosphoric acid is attained by the presence of a large amount of ammonium nitrate. The separation of molybdic oxide or iron salt is avoided by the low temperature employed.

For the final drying at 105° C. an air-bath was tried and decisively abandoned. The use of a liquid boiling at 108° C. to 110° C. is the only safe course. A water-oven consisting of several distinct divisions or floors, one above the other, and surrounded with dilute glycerol, 1.160 sp. gr. boiling at 110° C., was found to work admirably. The lower bath or division is reserved for the final drying. None but dry or almost dry precipitates must be allowed in this lower division. The precipitate may be dried in an ordinary water-oven almost to a constant weight and then dried to constant weight in a glycerol oven at 105° C. The filter papers used are dried at 105° C. and weighed between large, closely fitting ground watch-glasses. The final weighings of papers and contents are made in the same manner.

The following investigation serves to show the results that are secured by this method of analysis.

A chemically pure microcosmic salt was finely pulverised. Careful ignition of 10 grms. in a covered platinum dish gave 4.8955 grms. of sodium phosphate, giving a percentage by calculation of 34.07 per cent phosphorus pentoxide. Ten grms. of the salt were now dissolved in 1 litre of water and aliquots taken. Twenty-five c.c. containing 0.250 gm. of microcosmic salt were treated exactly as above. Fifty c.c. containing 0.500 gm. microcosmic salt were treated by the official magnesia method. The following results were obtained:—

	Taken.	Gladding method. Per cent.	Taken.	Magnesia. method. Per cent.
1	0.250	34.07	0.500	34.07
2	0.250	34.08	0.500	34.05
3	0.250	34.06	0.500	34.09
4	0.250	34.10	0.500	34.08

* Read before the New York Section of the American Chemical Society. From the *American Fertiliser*.

A solution of one-tenth of the above strength was obtained by dilution. Of this the following quantities were used and the phosphoric acid therein obtained by the new method:—

	Taken. c.c.	Yellow salt. obtained.	Phosphorus pentoxide obtained.	Theoretical phosphorus pentoxide.
1	10	0'091	0'00342	0'003407
2	1	0'010	0'00037	0'00034

These last experiments demonstrate the insolubility of the yellow salt and the applicability of the new method to very small amounts of phosphoric acid.

A number of comparative tests of fertilisers gave closely agreeing results, as follows:—

	Official method. Per cent.	New method. Per cent.
Phosphoric acid ..	28'80	28'87
Phosphoric acid ..	2'63	2'70
Phosphoric acid ..	12'03	12'00
Phosphoric acid ..	28'30	28'33
Phosphoric acid ..	15'64	15'70
Phosphoric acid ..	15'04	15'00
Phosphoric acid ..	15'19	15'23
Phosphoric acid ..	29'16	29'23

In all fertiliser work 0'250 grm. was used for precipitation, and molybdate solution to an excess of about 10 c.c. was added. No more than 10 c.c. in excess should be used.

Tankages and fertilisers containing a notable amount of organic matter should be ignited before solution.

An application of this method to the direct determination of reverted or citrate soluble phosphoric acid promises good results. The method of procedure is as follows:—The citrate filtrate and washings are made up to 200 c.c. Twenty-five c.c., equivalent to 0'250 grm. of the fertilizer are treated as follows:—Fifty c.c. ammonia, 0'900 sp. gr., are added and then nitric acid to acidity. The liquid is now diluted to half a litre to overcome the solvent action of the ammonium citrate, and heated in a bath to 65° C. Fifty c.c. of molybdate solution are added in a thin stream with stirring, and the whole digested for 30 minutes. The rest of the analysis is conducted precisely as in ordinary work, except that the filtrate is heated for thirty minutes longer at 65° C. The liquid should remain clear.

The bath used for drying the precipitates is made of very heavy copper, with double turned joints, the three divisions, or floors, one above the other, are of the following:—Inside dimensions, 10 inches wide, 7 inches deep, and 4 inches high.

The two lower divisions are surrounded by the glycerol on all sides except the front; the level of the glycerol reaches to the middle of the upper division. The upper division answers for the preliminary drying, the two lower for the final and complete desiccation.

The following directions for fertilisers will be found useful:—

For 0'250 grm. of each of the following fertilisers use the following amounts of molybdate of ammonia solution:—

	P ₂ O ₅ , present. Grm.	Use of molyb- date solution. C.c.
Tankage	0'020 to 0'040	25
Ground bone	0'060	40
Fish scrap	0'020	20
Rock phosphate (low grade)	0'065	40
Rock phosphate (high grade)	0'090	55
Acid phosphate (total) ..	0'030 to 0'040	30
Superphosphate (total) ..	0'035	25
Bone-black	0'090	55

All filtrates are to be carefully tested as directed. In filtering and washing the yellow salt, a suction pump is recommended. Two final washings (of a c.c. each) of alcohol will greatly lessen the time required for drying the precipitates.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING DECEMBER 31ST, 1897.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, January 10th, 1898.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 175 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Dec. 1st to Dec. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 175 samples examined by us during the month, all were found to be clear, bright, and well filtered.

The rainfall at Oxford during December shows a small excess, the recorded amount being 2'50 inches, against a thirty-years' average of 2'10 inches; the excess is therefore 0'40 inch.

The details of the rainfall are shown in the following table:—

Rainfall in Inches at Oxford, Month by Month, during the Year 1897.

	Actual fall.	Mean of 30 years.	Difference from the mean.
January ..	1'85	2'16	-0'31
February ..	2'41	1'76	+0'65
March ..	2'61	1'50	+1'11
April ..	1'95	1'66	+0'29
May ..	0'85	1'83	-0'98
June ..	1'70	2'68	-0'98
July ..	2'57	2'63	-0'06
August ..	3'94	2'32	+1'62
September..	2'25	2'75	-0'50
October ..	1'32	2'75	-1'43
November ..	1'15	2'10	-0'95
December ..	2'50	2'10	+0'40
	25'10	26'24	-5'21
			+4'07

It will be seen from this table that there has been a total deficit for the year of 1'14 inches.

Our bacteriological examinations of 248 samples have given the following results; we have also examined 41 other samples, from special wells, stand-pipes, &c., making a total of 289 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 25 samples) ..	805
New River, filtered (mean of 25 samples) ..	21
Thames, unfiltered (mean of 25 samples) ..	15,369
Thames water, from the clear water wells of five Thames-derived supplies (mean of 123 samples) ..	45
Ditto ditto highest	372
Ditto ditto lowest	0
River Lea, unfiltered (mean of 25 samples) ..	1268
River Lea, from the East London Company's clear water well (mean of 25 samples) ..	22

Table showing the Average Monthly Bacterial Variations during the Year.

Month.	Thames, unfiltered.	Five Thames-derived Companies, filtered.	New River, unfiltered.	New River, filtered.	River Lea, unfiltered.	River Lea (East London), filtered.
January	6409	30	1420	37	1460	20
February	9580	22	1589	20	1177	40
March	9187	33	1160	30	1080	22
April	3277	38	892	13	708	27
May	2937	40	388	44	791	279
June	73893	74	2005	69	66822	69
	105253	237	7454	213	72038	457
	17542	39	1242	35	12006	76
						Mean of 1st 6 months.
July	4791	83	754	44	2494	125
August	4574	51	3420	44	5846	64
September	98485	72	387	26	3168	36
October	3547	51	397	35	1588	39
November	27173	27	488	13	593	12
December	15396	45	805	21	1268	22
	153966	329	6251	183	14957	298
	25661	54	1041	30	2492	49
						Mean of 2nd 6 months.
	21601	46	1141	32	7249	62
						Mean of 12 months.

The accompanying table shows the average microbic variation for the year of the various London waters. Taken as a whole, it is highly satisfactory as regards the efficiency of combined storage and filtration in producing a water unexceptional for domestic use. On two occasions the bacteriological condition of the water in one of the clear water wells was above the average, the number of microbes rising to double what we regard as a proper, although severe, standard for the water supply of a city like London. This condition was, however, only temporary, and the microbes present were altogether of the harmless river kinds.

It must be kept in mind that we have bacteriologically examined 3249 samples during the past year, instead of only 348 examined in 1896. We now know from day to day the efficiency of each filter in use. The character of the supply of the respective Companies is therefore much more continuously and severely scrutinised than can possibly be the case when bacteriological examinations are taken at intervals of a week or a month. The result of this continuous examination is that we are in a position to immediately inform the Engineers of the Companies of the commencement of any deterioration in the action of the filters.

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

NOTICES OF BOOKS.

The Phase Rule. By WILDER D. BANCROFT. Ithaca N.Y.: *The Journal of Physical Chemistry*.

It is a little difficult to understand why the author has adopted the above title to his work. One's first impression on opening the volume is that Mr. Bancroft has, by dint of much research amongst the published accounts of original chemical work in many lands, evolved a new rule, or improved upon an old one, by which the nature of the pressure-temperature concentration curves of chemical systems could be predicted, together with the critical points on such curves, and that a comparison between various curves as predicted by his (or Gibbs's) rule, and the curves as plotted from the recorded experiments of various observers would be given. Such, however, is not the case.

The phase rule is simply a method of calculating the maximum possible number of branches to pressure-temperature concentration curves of a given chemical system when the total number of chemical compounds or "phases" which can be formed from the elements present is known, due allowance being made for the non-formation of certain compounds in many cases, owing to the presence of antagonistic elements in the system.

Now, since it can only be determined experimentally what compounds can or cannot be formed from a given collection of elements by suitably varying the pressure, or temperature, or concentration, or either or all of the independent variables, and the results of the investigation, when complete, can be plotted at once, and the whole curve with all its branches and critical points displayed; and since the data for calculating the number of branches is not complete until the investigation is far advanced, the "phase rule" appears to be redundant.

There is, however, another standpoint from which the contents of the book may be examined—one which is indicated by the author in the preface, viz., that it is a collection from many sources of all available information about recent research into the pressure-temperature concentration curves of chemical systems, classified according to the nature of the curves and the complexity of the system.

Viewed in this light the work has a distinct value, and we venture to express a hope that the author will from time to time publish supplements to the present volume.

The word "phase" is used as a substantive, and is defined to be "a mass chemically and physically homogeneous," or as "a mass of uniform concentration." Thus ice, water, and vapour are three phases, a solution of a salt, whether dilute or saturated, is a "phase," whilst if the salt begins to crystallise out of the solution there are two "phases," "salt" and "solution."

Chapter III. deals at great length with the pressure-temperature curves of water, sulphur, and phosphorus, these being taken as typical curves for a system of one component, and in this chapter occurs one of the few tentative forecasts that the author ventures upon, viz., that from the direction of the branches representing red phosphorus and vapour, and liquid phosphorus and vapour (page 32) it is just possible that red phosphorus, liquid phosphorus and vapour may co-exist in stable equilibrium at a very high temperature and great pressure, though he gives no idea as to the actual pressure and temperature probably required.

It is a pity the diagrams are not all drawn to scale with the meanings of the various branches attached, as is the case with a few in Chapters V. and VII.; if this were done in future editions the book would be very much easier to read, and it would not be necessary to refer back so often in order to understand the text; in fact a great deal of the text might be very well omitted if the diagrams were perfected.

Fig. 13 on page 79, a concentration temperature curve for H_2O and Fe_2Cl_6 , is one of the best in the book, and shows very clearly, without referring to the text, the various changes that take place in a solution of ferric chloride, as the concentration is varied from H_2O at 0° to Fe_2Cl_6 at 100° , beyond which point the curve has not been carried.

Chapters III. to X. deal with the curves for systems with two components only, these can all be clearly shown as plane curves on an ordinary sheet of squared paper.

Chapters XI. to XIX. show the principal types of curves in systems with three components; here, again, the pressure-temperature curves can be plotted without trouble as plane curves, but the concentration curves, "isotherms" and "isobars" require different treatment, and must either be shown as solid curves in three dimensions, or by one or other of the devices due to Schreinemakers, Meyerhoffer, Stokes, Roozeboom, &c.

Roozeboom's, the method adopted by the author, is the most satisfactory hitherto devised, though it requires a different diagram for every pressure to show a complete set of isothermal curves when the concentration of the system is varied, and is of little use in forecasting results, it is a perfectly clear record of results experimentally obtained, and, like all other curves, is of great use in detecting experimental or clerical errors.

Very few systems with three components have been systematically examined, and none have been exhausted, consequently the isothermal curves in this section of the book are generally largely guesswork sketches, whilst the author makes no reference to the companion series of isobars on a concentration-pressure diagram.

The first nineteen chapters, besides the curves which have been selected as types for illustration, contain numerous tables from which the corresponding curves for other systems may be wholly or partially plotted. A critical examination of these shows us what a large field yet lies open to original research.

Chapter XX. deals with systems of four components, limited by the proviso that one of the four is water, because no others have yet been examined.

In this class the curves would have to be so specialised and multiplied that the author has wisely made no attempt to shew any, and relies altogether upon the old-fashioned tables of figures, which after all can, with time and patience, be made to yield a great deal more information than is visible on the surface.

The amount of labour bestowed on this work by the author may be gauged by the fact that the published works (in nearly every European language) of no less than 200 writers have been consulted, and full references given for the benefit of those who may wish to consult the originals.

a priori reasoning is $\frac{3T}{ML} = \text{constant}$; T being the abso-

lute temperature of fusion, M the molecular weight, and L the heat of liquefaction per unit mass. Substituting $\frac{6.4}{S}$ (S = specific heat) for M, the relation $TS = 2.1L$ at

once obtains; this is the relation proposed in different forms by Dr. Richards, Mr. Crompton and myself, and by me restricted as holding only between elements closely related to each other. The value of the ratio between the heat of liquefaction and the total heat at the melting point obtained above is of the same order, but smaller than the empirically derived numbers given by me; the reason of this is supplied by Mr. Beveridge's argument introducing the polymerisation of the molecule in the liquid state.—I am, &c.,

NOEL DEER.

Windsor Forest, West Coast, Demerara.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Seances, de l'Académie des Sciences. Vol. cxxv., No. 23, December 13, 1897.

The Minister of Public Instruction communicated the approval of the President of the Republic of the election of M. Ditte as a member of the Chemical Section, in the place of the late Prof. Schützenberger.

New Method for the Attack of Platinum. Preparation of Bromoplatinate of Ammonium and of Potassium.—Georges Méker.—Already inserted.

Phosphorous Oxide.—A Besson.—Already inserted.

Properties of Carbide of Sodium.—Camille Matignon.—Carbide of sodium is obtained in the form of a white powder; its density at 15° is 1.575, and it appears to be quite insoluble. Dry air and oxygen have no effect on it at ordinary temperatures, but on gently heating combustion takes place, leaving a residue of Co_2Na_2 . In the presence of chlorine gas it becomes incandescent, and with bromine the reaction is of almost explosive violence. Iodine has a more moderate action, and CaI_4 , melting at 185° , can be obtained. Hydrogen has no action at all. When thrown into water, carbide of sodium explodes violently, giving a deposit of carbon. It also becomes incandescent when in contact with CO_2 and SO_2 . It acts in the cold on a large number of organic substances. The primary and secondary alcohols give off acetylene, giving rise at the same time to the corresponding alcoholate $2C_3H_7OH + C_2Na_2 = C_2H_2 + 2C_3H_7ONa$.

Series of New Cyclic Ketones.—A. Behal.—These ketones are obtained from the heavy oils distilled from Stockholm tar, by first treating them with alkalis; this removes the acids, phenols, &c. The supernatant oil, after washing with distilled water, is shaken up with hydrochloric acid, in proportions variable according to experience, until when diluted and neutralised with carbonate of soda, none remains unacted upon. This solution is treated with a current of steam, and the water being distilled off, leaves an oily layer which is decanted. After repeating this operation once or twice, a slightly yellow liquid, smelling of menthol, remains. The ketones are separated from this liquid by means of hydrochloric acid of variable strengths. To obtain the ketones in a state of purity, they are transformed into oximes by means of hydrochlorate of hydroxylamine and oxide of zinc, in alcoholic solution. The benzoyl derivative is separated in the solid state by treating the oxime with chloride of benzole in the cold. When the

CORRESPONDENCE.

MOLECULES AND LIQUEFACTION HEATS.

To the Editor of the Chemical News.

SIR,—The letter (from Mr. P. J. Beveridge appearing in the CHEMICAL NEWS (vol. lxxvi., p. 264) is of interest as giving a theoretical basis to one of the relations discussed by me (CHEMICAL NEWS, vol. lxxvi., p. 234). Expressed symbolically, the relation obtained by Mr. Beveridge on

benzoyle are treated in alcoholic solution with an excess of soda, the oximes are easily regenerated in a state of great purity. The oxime fusible at 102° furnishes a ketone boiling at 192° ; it is soluble in water, and its density at 0° is 0.9539. These ketones are very numerous, and belong for the most part to the series of benzenic tetrahydrides, which are at present very little known.

Neutralisation of Glycero-phosphoric Acid by the Alkalies in the presence of Heliantine A and Phenolphthalein.—H. Imbert and A. Anstruc.—Experiments have shown that, contrary to expectation, glycero-phosphoric acid does not act on heliantine A and phenolphthalein in the same manner as phosphoric acid. It first behaves as an acid, but if a sufficient quantity of soda be added to render it neutral to heliantine, it still remains acid to phthalein.

Heat of Neutralisation of Glycero-phosphoric Acid.—H. Imbert and G. Belugou.—The authors give a table showing the heat of neutralisation of glycero-phosphoric acid, with both soda and potash, from which it appears that the first molecule of the alkali gives off as much heat as when acting on free phosphoric acid; the second molecule gives off considerably less heat, while the action of the third molecule is nil.

No. 25, December 20, 1897.

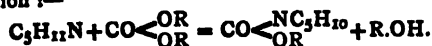
The Permanent Secretary announced the loss the Academy has sustained by the death of M. Brioschi, of Milan.

Duration of the Phosphorescent Power of Sulphide of Strontium.—Jose Rodriguez Mourello.—Five tubes containing sulphide of strontium, prepared either—A, by reducing sulphate of strontium with carbon; B, from carbonate of strontium and sulphur; C, by the action of sulphuric acid and strontia; D, by M. Verneuil's method; and E, by M. Verneuil's method modified by the author (M. Mourello). They were first kept in the dark for three days, so as to entirely lose all phosphorescence; then, after being simultaneously exposed to light for 15 minutes, they were again taken into the dark, and the relative brightness of each was in accordance with the order in which they are here mentioned, A, B, C, D, E. After an hour, A was much less bright, B had lost a little in intensity, but C D and E showed no change; fifteen minutes later C began to show a difference, followed shortly afterwards by D, while E remained still without any apparent change. A and B had lost all their phosphorescence three hours later, C after five hours, and D after six hours, whilst E still retained some phosphorescence after 12 hours, though only to a very small extent.

Volumetric Estimation of Antimony.—H. Causse.—The author describes a new method for the volumetric estimation of antimony, which is based on the fact that when antimonious acid, either free or combined, comes into the presence of iodic acid, this latter is destroyed, and the antimonious acid passes integrally to the state of antimonie acid, while a quantity of iodine proportional to the amount of the iodic acid decomposed is set at liberty, $5\text{Sb}_2\text{O}_3 + 2\text{IO}_3 = 5\text{Sb}_2\text{O}_5 + 2\text{I}_2$.

On Cerium.—M. Boudouard.—(See p. 27).

Action of Piperidine on the Carbonic Ethers from the Phenols; Formation of Aromatic Urethanes.—MM. Caseneuve and Moreau.—Piperidine, unlike the primary aromatic amines, does not give a symmetrical ureate, but always a urethane, according to the following equation:—



R being an aromatic radical. This reaction recalls the action of a molecule of ammonia on carbonate of ethyl, but with piperidine the reaction is limited to a urethane. Three hitherto unknown urethanes are described, viz., phenyllic, galacolic, and naphtholic B urethanes from piperidine.

MEETINGS FOR THE WEEK.

- MONDAY, 24th.—Society of Arts, 8. "Decorative Bookbinding," by Cyril Davenport.
TUESDAY, 25th.—Society of Arts, 8. "Renaissance Woodwork in England," by J. Hungerford Pollen.
— Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
WEDNESDAY, 26th.—Society of Arts, 8. "Fireproof Construction of Domestic Buildings," by Thomas Potter.
THURSDAY, 27th.—Royal Institution, 3. "The Halogen Group of Elements," by Prof. Dewar, M.A., LL.D.
FRIDAY, 28th.—Royal Institution, 9. "Instinct and Intelligence in Animals," by Prof. C. Lloyd Morgan, F.G.S.
SATURDAY, 29th.—Royal Institution, 3. "Cyprus," by Prof. Patrick Geddes, F.R.S.E.

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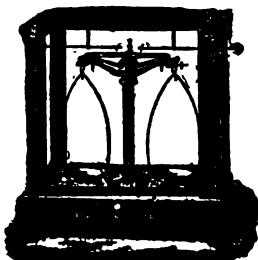
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CONTENTS.

ARTICLES:—	PAGE
A Note on some further Determinations of the Dielectric Constants of Organic Bodies and Electrolytes at very Low Temperatures, by James Dewar, M.A., LL.D., F.R.S., &c., and J. A. Fleming, M.A., D.Sc., F.R.S., &c.	37
Scorification versus Cupellation, by H. N. Warren	39
Persulphuric Acid and its Salts, by M. Elbe	39
On the Reactions between Mercury and Concentrated Sulphuric Acid, by Charles Backhouse and F. W. Miller	40
The Analysis of Bearing-metal Alloys, with a New Volumetric Method for Determining Copper, by W. E. Garrigue	41
NOTICES OF BOOKS.—Notes on Lead and Copper Smelting and Copper Converting—Chemistry for Photographers—The Production of Precious Stones in the United States in 1896—The Commercial Uses of Coal-gas—&c.	43
CHEMICAL NOTICES FROM FOREIGN SOURCES	44
MISCELLANEOUS	46
MEETINGS FOR THE WEEK	47
NOTES AND QUERIES	47

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A NOTE ON

SOME FURTHER DETERMINATIONS OF THE DIELECTRIC CONSTANTS OF ORGANIC BODIES AND ELECTROLYTES AT VERY LOW TEMPERATURES.*

By JAMES DEWAR, M.A., LL.D., F.R.S., Fullerian Professor of Chemistry in the Royal Institution, and

J. A. FLEMING, M.A., D.Sc., F.R.S., Professor of Electrical Engineering in University College, London.

(Concluded from p. 27).

SUBSEQUENTLY to the completion of the experiments described in this paper the suggestion has been made by R. Abegg (*Wied. Ann.*, liii., p. 249) that the high dielectric values at low temperatures are due to polarisation of the electrodes of the condenser, and that the capacity measured is a polarisation capacity and not a true dielectric capacity, and he supports this contention by pointing out that whenever we have obtained a large dielectric value at the low temperature it has always been measured with an electromotive force of 1.434 volts, which is less than the ordinary full reverse electromotive force of polarisation.

There are, however, reasons for considering that this contention is not a valid one. In the first place, we have always in all the measurements begun operations by testing the dielectric capacity of our condenser with an electromotive force of one Clark cell (= 1.434 volts) in order to see roughly whether the dielectric value was large or small. If it was a small value we then gradually increased the electromotive force until a good readable galvanometer deflection was obtained. We never found that, with an electromotive force of 1.434 volts, the dielectric constant of any substance was greater than with a much higher voltage.

In the next place, in many cases we changed from a working electromotive force of about 20 volts to one of 1.434 volts, and we never found any marked discontinuity in the calculated value of the dielectric constant at that point. If our previous papers on this subject are examined the following instances may be found.

TABLE VII.—Measurement of various Dielectric Constants with different Electromotive Forces.

Substance.	Temperature.	Dielectric constant.	Voltage with which constant was measured.	Reference.
Ice	- 89.4	27.6	19.8	<i>Roy. Soc. Pr.</i> , lii., p. 318.
	- 87.2	29.0	1.4	
	- 124.2	18.1	20.3	
"	- 119.2	21.8	1.4	<i>Ibid.</i> , p. 321.
	- 118.5	21.2	20.2	
	- 114.0	22.3	1.4	
Sodic chloride, 10 p.c. solution ..	- 112.4	16.9	20.0	<i>Ibid.</i> , p. 307.
Potassic chromate, 30 p.c. solution ..	- 100.0	22.3	1.4	
Cupric carbonate, 10 p.c. suspension	- 124.7	16.5	18.2	
Baric hydrate, 5 p.c. suspension	- 120.8	21.8	1.4	<i>Ibid.</i> , p. 390.
	- 178.0	23.9	19.5	
	- 174.2	25.5	1.4	
Bismuth oxide, 10 p.c. suspension	- 129.2	19.9	17.8	<i>Ibid.</i> , p. 372.
	- 127.3	24.5	1.4	

* A Paper read before the Royal Society, December 9th, 1897.

An examination of the above instances will show that if the electromotive force is changed from about 20 volts to 1.4 volts, it does not make a greater difference in dielectric constant than can be properly ascribed to the accompanying change in temperature.

[Again the following measurements were made at about the same voltage :—

Substance.	Temperature.	Dielectric constant.	Charging voltage.
Potassic bicarbonate ..	- 166.5	2.80	19.8
Sodic bicarbonate ..	- 166.7	48.70	19.8
Ferric chloride ..	- 133.8	4.23	19.8
Sodic chloride ..	- 129.2	14.55	20.2
Cupric carbonate ..	- 132.7	3.42	18.2
Cupric sulphate ..	- 133.2	16.40	19.7
Lithic hydrate ..	- 198.0	3.23	19.8
Baric hydrate ..	- 196.8	20.10	19.5

It is unlikely that polarisation accounts for the differences between the dielectric constants of the above substances taken, pair and pair, when measured at nearly identical temperatures, and very nearly the same voltages. —November 8, 1897.]

In order to settle the matter finally we propose, however, to re-measure a number of those substances which have shown high dielectric values at the low temperature when measured by the galvanometric method at a frequency of 120, but using in all cases an electromotive force of 100 volts.

If under the larger electromotive force the dielectric values of some electrolytes still remain large, it will be difficult to ascribe this large value to polarisation.

The facts, however, admit of another interpretation. It is clear that the dielectric constants of some substances at low temperatures are vastly more susceptible to change of electromotive force frequency than is the case with others, and that the electric strain produced by a given electric stress varies in some cases enormously with the time of imposition of the stress but very little in others.

Another argument against the view that these high dielectric values are due to polarisation, as ordinarily understood, is as follows :—The results of most numerous experiments on water show that the dielectric constant at or near 0° C. is a number not far from 80. This value is obtained whether the electromotive force reversals are infinitely slow or whether they are very large. The results of the measurement of the electrical refractive index of water even with ether waves only 4 m.m. in length, and, therefore, having a frequency of about 7.5×10^{10} , as given recently by Lampa (*Wien. Ber.*, Part 2a, p. 587, 1896; also p. 1049, 1897), indicate a number not far from 9.5 as the refractive index, and hence give a dielectric value of 90. There can be no question of polarisation of electrodes in this last case. On the other hand, an increase in frequency which hardly affects the value of the dielectric constant of water is sufficient to greatly decrease that of ethylic alcohol, and at the same frequency the dielectric constant of ethylic alcohol was found by Lampa to have fallen to a value of 5. Hence ethylic alcohol is more sensitive to change of frequency than water. There is, therefore, no *a priori* reason why we might not expect to find the same thing even in a much greater degree in certain other bodies at lower temperatures, viz., a true high dielectric value for a certain frequency, but great sensitiveness to increase of frequency in such fashion that increase of frequency greatly reduces the dielectric value. At the present stage of the enquiry it seems undesirable to endeavour to regard the facts wholly from the point of view of one hypothesis as to the nature of electrolysis.

We have again to mention with pleasure the assistance we continue to receive from Mr. J. E. Petavel in the observational part of these investigations.

Note added December 8, 1897.—Since the above paper was printed we have repeated some of our former experi-

ments with the 5 per cent solution of rubidic hydrate and the 5 per cent solution of potassic hydrate, using the original method in which a condenser having the frozen electrolyte as dielectric is charged and discharged through a galvanometer, but employing much higher charging voltages. The object of these experiments was to apply a further test of the validity of the contention put forward by R. Abegg, that we have obtained high dielectric values for certain of these frozen electrolytes in consequence of having invariably used an electromotive force of 1'434 volts in the experiments with these particular substances. In order to be able to work with larger electromotive forces we arranged three galvanometers of the Holden d'Arsenval type otherwise exactly similar, except in having different sensibilities and resistances. One was the 500-ohm galvanometer used in all our previous condenser experiments, another was a 100-ohm coil galvanometer, and a third was a 4-ohm coil galvanometer. These were used at the same distance (125 c.m.) from the scale as formerly. An approximate test for the relative sensibility of these galvanometers was made by placing a Clark standard cell in series with each galvanometer through 100,000 ohms and noting the scale deflection produced. As the internal resistance of the galvanometers was at most only $\frac{1}{2}$ per cent of the total resistance, these scale deflections may be considered to be approximately produced by the same current. The scale deflections in centimetres were—

For the 500-ohm galvanometer..	37'3 c.m.
" 100-ohm "	.. 6'7 "
" 4-ohm "	.. 0'55 "

Hence the sensibilities are in the ratios of these deflections, and the deflections of the 100-ohm galvanometer must be multiplied by 5'5, and those of the 4-ohm galvanometer by 67'8, to reduce their scale readings to equivalent deflections in terms of the 500-ohm galvanometer.

The 500-ohm galvanometer was then used with the condenser and vibrator, as described in one of our previous papers (*Roy. Soc. Proc.*, lxi., p. 300, 1897). The condenser having gaseous air as dielectric, the scale deflection for a frequency of 120 and an electromotive force of 97 volts was 3'2 c.m. when corrected for capacity of leads.

The condenser then had its dielectric space filled with the 5 per cent solution of rubidic hydrate, and was frozen in liquid air. The dielectric constant was next measured, using an electromotive force of 17'8 volts and the 500-ohm galvanometer. The value of the dielectric constant found, when corrected for the capacity of the leads, was 65'6. The mean corrected scale deflection was 38'5 c.m. for 17'8 volts. This, reduced to its equivalent for 97 volts, is 210 c.m. and $210/3'2 = 65'6$.

In the next place, the same experiment was repeated employing the 100-ohm galvanometer and an electromotive force of 79 volts. Applying the necessary corrections to the observed scale deflection of 23'5 c.m., and reducing to the equivalent deflection on the 500-ohm galvanometer gave 160 c.m. as the value of the reduced deflection. Hence $160/3'2 = 50$ is the dielectric constant. The rather considerable difference between these values (65'6 and 50) is not a matter for surprise, having regard to the extreme steepness of the dielectric curve of the rubidic hydrate solution at about the temperature of liquid air. As we were merely desirous of determining whether a considerable increase of electromotive force would greatly diminish the large dielectric value, we did not trouble to put in operation the rather elaborate platinum thermometer arrangements for determining the exact temperature of the dielectric. A reference to the dielectric-temperature curve of rubidium hydrate (*Ibid.*, p. 378) will show that even one or two degrees of temperature change in the neighbourhood of -185°C. , or -200°pt. makes a very considerable alteration in the dielectric value. The result, however, ascertained is that changing the electromotive force from 1'434 volts to 17'8 or 79 volts does not bring down the dielectric constant from a large value to a small one.

In the same way the 5 per cent solution of potassic hydrate was tested.

Using the 500-ohm galvanometer and an electromotive force of 9'88 volts we found 153 as the dielectric constant of the frozen electrolyte at the temperature of liquid air. Employing the 4-ohm galvanometer and 79'5 volts we found 175 as the dielectric constant.

All the above observations were taken at the temperature of liquid air and with an electromotive force frequency of 120. It is, therefore, quite clear that as far as these two frozen electrolytes are concerned, raising the charging voltage to a value far above that of the average electromotive force of polarisation does not bring down these abnormal values of the dielectric constant. On the other hand, a relatively small decrease in the temperature or increase in the frequency at low temperatures suffices to reduce the dielectric value of these frozen hydrates very considerably.* We may, therefore, say that the contention put forward by R. Abegg that the high dielectric values we have found for certain substances at the liquid air temperature are really polarisation capacities, does not seem to be borne out by the results of further experiment, and for the following reasons:—

- i. Because a very great increase in the charging electromotive force does not in any corresponding degree reduce the abnormally high dielectric values of certain frozen electrolytes to much smaller values.
- ii. Because when, in the course of observations to construct a temperature dielectric curve, the working electromotive force has been changed from a value below the counter-electromotive force of polarisation to a value far above it, there is no break or discontinuity in the curve of dielectric value.
- iii. Because the great difference between quite similar electrolytes, such as the 10 per cent solution of potassic and sodic carbonates, in respect of dielectric constant at equally low temperatures and under equal charging electromotive forces, is left unexplained.
- iv. Because in the case of many substances, such as frozen ammoniac hydrate, ice, and oxide of copper in suspension in ice, at very low temperatures, we find high dielectric values, even though employing alternating currents of fairly high frequency (350 ω).
- v. Because the high dielectric value of water and alcohol and other bodies at ordinary temperatures remain, even when the observations are taken, with alternating electromotive forces of exceedingly high frequency and under conditions when there are no electrodes to polarise, as when the electric refractive index is measured with electromagnetic radiation.

We consider that the results of observations so far made are best expressed by merely considering the dielectric constant to be a function of the frequency and the temperature, and represented therefore by a *dielectric surface*, which surface has for some substances a region of abnormal dielectric ordinate.

In all cases so far examined by us, lowering the temperature sufficiently acts in the same manner in reducing the dielectric constant as sufficiently increasing the frequency, and both actions reduce the abnormally large dielectric values of some substances to values more approximately equal to the square of the optical refractive index of the body.

* The effect of increased frequency of electromotive force reversals in decreasing the dielectric constant is evidently dependent upon the temperature as well as on the physical state of the body. In the case of water an increase in the frequency from zero to 10^9 hardly affects the dielectric constant at all. In the case of ice at 0°C. the same increase in frequency reduces the dielectric constant from 80 to between 2 and 3. In the case of ice at -50°C. , as we have shown above, an increase in frequency from 120 to 350 reduces the dielectric constant from about 60 to about 3'6. (December 21, 1897).—A. W. R.

The question then to be considered is the physical reason for the high dielectric values for particular substances for certain ranges of electromotive force, frequency, and temperature. Whether this abnormal electric displacement is considered to be the result of a molecular strain superimposed on a true electric strain, or whether it is the beginnings of that molecular deformation which finally ends in chemical decomposition, remains to be seen. Having regard to the enormously high electrical resistivity which we have shown these frozen dielectrics to possess, it does not appear to us likely that polarisation in the sense of a deposition of ions on the electrodes can be invoked to explain the differences we have shown exist.

SCORIFICATION VERSUS CUPELLATION.

AN IMPROVED METHOD FOR LABORATORY PURPOSES.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

IN order to carry out a successful cupellation by the aid of the well-known muffle furnace, especially when results of great accuracy are sought for, many various precautions have to be considered, both as regards the careful regulation of temperature and also with respect to the amount of air admitted into the furnace, so as to continuously produce a superficial oxidation of the lead to litharge, as fast as absorbed by the bone-ash comprising the cupel. On the other hand, scorification of a regulus produced from the smelting of argentiferous lead is often resorted to prior to cupellation of the same, in order to reduce in bulk the original matrix, and by so doing at least partially avoid the more tedious operation of cupellation. Again, the addition of alkaline nitrates is frequently adopted in order to provide a quick oxidant prior to cupellation, although in most cases affording inaccurate results. In order, therefore, to obviate these difficulties, the author has for some time past employed both strontium and barium sulphate as a substitute for bone-ash, at the same time avoiding entirely the tedious process of cupellation; and, by so doing, arrives at more accurate results in a very short space of time. The process is conducted in the simplest manner possible by the introduction into an ordinary clay roasting-dish of convenient dimensions of a sufficiency of barium sulphate; a small bed being sunk into the centre of the same by gently compressing the powder by the aid of a smooth pebble.

The cavity thus formed is now quickly raised to a dull red heat by means of an ordinary blowpipe attached to a foot bellows. The alloy is next dropped in, to which is immediately added the same bulk of a further alloy, consisting of lead in admixture with 10 per cent of bismuth. The flame from the blowpipe is now allowed to impinge upon the melt until it assumes a bright red tint; the flame is then shortened and raised to within half an inch of the same, and a full blast of air maintained. The regulus at once assumes a spherical condition and commences to rotate rapidly, at the same time disappearing with remarkable rapidity. About one-quarter of the litharge thus oxidised is absorbed by means of the sulphate, the remainder solidifying around the lead.

In cases where large quantities are employed, the operation should be stopped at intervals, and the halo thus formed removed by a spatula; the scorification is then continued until pure silver remains.

The speed with which the lead is thus oxidised and removed by means of the bismuth alloy is most remarkable. By means of a barytes bed only 6 inches in diameter I have frequently removed as much as 2 lbs. of lead in less than an hour. The facility also of obtaining the sulphate in most laboratories where estimations of sulphur are carried out will be easily recognised. If a sufficiency, however, cannot be thus obtained, the com-

mercial ground mineral (which answers the purpose admirably) can always be relied upon in any quantity and at a very low rate.

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PERSULPHURIC ACID AND ITS SALTS.

By M. ELBS.

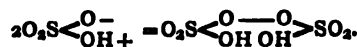
IT is hardly necessary to recall the fact that persulphuric acid was obtained for the first time by Berthelot in 1881, by the electrolysis of ordinary sulphuric acid. The properties of this acid have been since studied by Richarz (in 1888), and again in 1891 by Marshal, who also described some of its salts.

Since 1893 the work done on this acid has considerably increased, and it seems to me to be of some value to give a review of what has been done.

Aqueous sulphuric acid is a conductor of electricity; and, further, a conductor of the second class, since, while allowing the current to pass, it becomes decomposed, giving two distinct products at the two poles. Its electrical dissociation furnishes theoretically hydrogen and an acid remainder. But as sulphuric acid, in its quality of a bibasic acid, contains in its molecule two atoms of hydrogen, its dissociation can be effected in two distinct manners:—

I. *In a Complete Manner.*—Each molecule of H_2SO_4 gives two ions (H^+) and one bivalent ion (SO_4^{2-}) according to the equation $\text{H}_2\text{SO}_4 = 2(\text{H}^+) + (\text{SO}_4^{2-})$. Very dilute solutions of sulphuric acid behave in this manner. But, as a matter of fact, by electrolysis they always give off a gas, $\text{H}_2 + \text{O}$. The remainder (SO_4) goes to the positive pole, and reacts on the water according to the equation $\text{SO}_4 + \text{H}_2\text{O} = \text{H}_2\text{SO}_4 + \text{O}$, while at the negative pole we obtain the two corresponding atoms of hydrogen; or, to be more precise, the sulphuric acid always remains as sulphuric acid, and it is the water only which is dissociated into its elements H_2 and O .

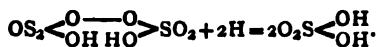
II. *In an Incomplete Manner.*—A more concentrated sulphuric acid ($D = 1.4$) gives by electrolysis the ions (H^+) and (HSO_4). The ions (HSO_4) go to the positive electrode and combine mutually, at any rate to a great extent, and form persulphuric acid:—



As a secondary reaction these same ions may react to a small extent on the water of the electrolyte, and re-form sulphuric acid with liberation of free oxygen:—

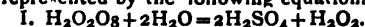


At the negative pole two atoms of hydrogen are given off. According to these reactions, dilute solutions of sulphuric acid (density lower than 1.2) will not give any persulphuric acid at all, while solutions of medium concentration (densities from 1.3 to 1.5) of which the ions may be represented by (H^+) and (HSO_4), will in almost every case give persulphuric acid. And, in the latter case, the proportion may reach 70 per cent of the theoretical amount. For higher concentration the return diminishes. Firstly, because the concentrated sulphuric acid may partially decompose the persulphuric acid, and secondly, because the latter is a bad conductor of electricity. In contact with the negative pole, persulphuric acid should be decomposed by the hydrogen which is disengaged:—



It therefore becomes necessary, in order to obtain good results, to separate the liquid round the anode from that round the cathode by means of a porous pot.

Free persulphuric acid is very unstable in aqueous solution. According to the conditions, it will decompose into sulphuric acid and peroxide of hydrogen, or sulphuric acid and oxygen. These two forms of decomposition may be represented by the following equations:—

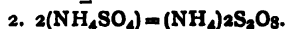
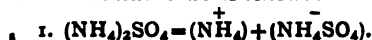


When warm the decomposition takes place only according to the second equation.

The principal reactions of persulphuric acid are as follows:—

1. Disengagement of oxygen by heat.
2. Decolouration of indigo.
3. Decomposition of hydrochloric acid or chloride of sodium, with the production of chlorine, and the analogous reaction with bromide and iodide of potassium.

Among the salts of persulphuric acid, the best known is the persulphate of ammonium, which can be prepared by the electrolysis of a saturated solution of sulphate of ammonia. The reactions are as follows:—



Persulphate of ammonium takes the form of white crystals. It is stable when in the dry state, and up to the temperature of 100°C ., but, on the other hand, it decomposes when moist, even at the ordinary temperature, with the production of strongly ozonised oxygen:—



Two parts of cold water dissolve one part of persulphate of ammonium. Persulphate of ammonium gives the same reactions as persulphuric acid, besides some others, as follows:—

1. When warmed with a solution of sulphate of aniline, it gives aniline black.
2. A solution of fuchsine with acetate of soda is decolourised by persulphate of ammonium.
3. With a solution of manganous sulphate, persulphate of ammonium gives a precipitate of binioxide of manganese. This reaction is expressed by the following equation:—

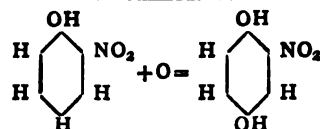


4. With a solution of carbonate of potassium, persulphate of ammonium gives a very dense, crystalline precipitate of persulphate of potassium. No salt of persulphuric acid is insoluble. Persulphate of potassium is only difficultly soluble, but all other persulphates dissolve easily.

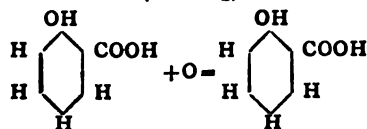
The slight solubility of persulphate of potassium enables one easily to prepare this salt by the electrolysis of a cold saturated solution of sulphate of potassium in sulphuric acid of 1.3 density. Under these conditions, and using a current of about three ampères, we can rapidly obtain a crystalline deposit of persulphate of potassium.

The metallic sulphates, treated under the same conditions, behave in a very variable manner. Those which give the best return in persulphate are those of ammonium, potassium, and aluminium. Persulphate of ammonium only, up to the present, seems destined to be of any industrial use. Its use as a bleaching agent is already under consideration, and it is already being used for the purpose of introducing directly the hydroxyl group into the benzinic nucleus.

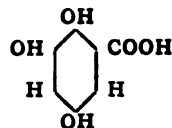
Thus ortho-nitrophenol in alkaline solution, treated with persulphate of ammonium, becomes oxidised and produces nitro-hydroquinone:—



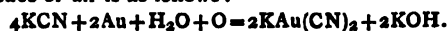
In an analogous manner salicylic acid would be transformed into hydroquinone, carbonic acid, or into pyrocatechine carbonic acid (Schering).



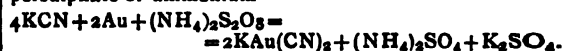
or else—



In the same manner we can transform oxyanthroquinone into alizarine, alizarine into purpurine, Bordeaux alizarine, or alizarine cyanine. Persulphate of ammonium is also used, concurrently with cyanide of potassium, for the extraction of gold. The ordinary reaction in the presence of air is as follows:—



Oxidation is much more rapid when we make use of persulphate of ammonium—



It seems very probable that the applications of persulphate of ammonium will become more and more numerous, the more so as the cost of its manufacture is low. But at the same time it must not be forgotten that as an oxidising agent it is inferior to many other substances in common use. Its decomposition only furnishes one molecule of free oxygen, while bichromate of potassium, for example, gives three.—*Zeitschrift für Angewandte Chemie*, p. 195, 1897.

ON THE REACTIONS BETWEEN MERCURY AND CONCENTRATED SULPHURIC ACID.

By CHARLES BASKERVILLE and F. W. MILLER.

MANY text-books class the reactions between copper and concentrated sulphuric acid along with mercury and sulphuric acid, stating merely that a sulphate of copper or mercury is formed with the evolution of sulphur dioxide.

In other communications (*Four. Am. Chem. Soc.*, xvii., 904, and xviii., 942), one of us (Baskerville) has shown that while the formula—



expresses in fact the main reaction when these two substances are brought together, even at different temperatures, that only at 270°C ., or higher, is that reaction unaccompanied with very important and complex secondary reactions. In preparing sulphur dioxide, by heating together copper and concentrated sulphuric acid, invariably a black or brown material discolours the colourless acid. This dark substance is, in the main, cuprous sulphide.

Not finding the reaction between mercury and concentrated sulphuric acid worked out in detail in any of the literature at our command, and since so much wrong data have been recorded concerning copper under similar conditions, it was deemed advisable to study this reaction at different temperatures,

The reaction is by no means like that of copper. The primary reaction depends upon the preponderance of mercury or acid, the temperature, and the time during which it is allowed to continue.

If the mercury be in larger amounts than the acid, invariably almost pure mercurous sulphate is produced:—



It will be well to state here that the mercurous sulphate so produced is a white crystalline substance and not black, as stated in Roscoe and Schorlemmer's "Treatise on Chemistry" (vol. ii., Part i., p. 401).

Series of experiments in duplicate were carried out at different temperatures when the acid was in large excess. A qualitative experiment showed that mercury decomposed concentrated sulphuric acid at the ordinary atmospheric temperature, about 20° C. This experience is contrary to that stated in a number of text-books. Experiments carried out at 100° C. upon a water-bath, lasting during twenty hours, in apparatus especially arranged to insure only the presence of dust-free, dry air showed that about one-fifth of the mercury had entered into the reaction. Ten grms. of the mercury and from 50 to 100 grms. acid were used. Expediting the results to exhibit this reaction:—



we were surprised to find so much less sulphur dioxide evolved than should have been. The crystalline sulphate produced gave 20.75 per cent SO_4 , which is very near 19.35 calculated for mercurous sulphate. Again, at 150° C., when the flask containing the mercury and acid was kept at a practically constant temperature by being immersed in a sulphuric acid bath for two hours, the amount of sulphur dioxide evolved was much below the theoretical. At least half of the mercury in each of these experiments was dissolved. The white crystalline layer of sulphate which separated out as the amount of metallic mercury decreased was augmented by another layer, which had been in solution, when the acid became cool. Both were crystalline, but while the latter was powdery, the former was composed of needle-like crystals. To avoid the formation of the basic sulphate, $\text{HgSO}_4 \cdot 2\text{HgO}$, which was always produced in our efforts to wash out the remaining acid with water, we used ninety-five per cent alcohol. Four washings were sufficient; in fact, on further washing even with alcohol, some basic sulphate was formed. On analysis of this alcohol-washed substance, which had been dried to a constant weight at 100° C., we obtained:—

	Calculated for $\text{Hg}_2(\text{SO}_4)_2$ or $\text{Hg}_2\text{SO}_4 \cdot \text{HgSO}_4$.	Found.
SO_4	24.24	23.96 24.27
Mercury (-ous) ..	50.38	50.27

At 200° C. the percentage of mercurous mercury dropped to 40; at 250° C. still lower, showing a steady decrease in the amount of mercurous sulphate produced as the temperature increased. When the acid was boiled and the mercury poured in, the crystalline compound gave 32.91 per cent of sulphur tetroxide when mercuric sulphate contains 32.43 per cent. Evidently some of the acid was not removed by the washing with alcohol.

It was desirable to determine whether the primary reaction took place in steps, *e.g.*, if the metal liberated hydrogen, which in the nascent state attacked the remaining sulphuric acid with the production of sulphur dioxide, such having been recorded by some authors to be the case in the reaction between copper and sulphuric acid. Careful experiments at different temperatures, 150° and 200° C., were carried out in an atmosphere of carbon dioxide free from air. One hundred c.c. of the gases given off and collected over a ten per cent solution of sodium hydroxide gave not a trace of hydrogen. The sulphuric

acid in this case is evidently reduced directly by the metal. No sulphide was produced, nor any free sulphur.

If portions of the crystalline compound, produced by the treatment of mercury with concentrated sulphuric acid rendered free of acid by washing with alcohol and dried at 100° C., be again treated with acid in similar apparatus, sulphur dioxide is evolved in proportion to the amount of mercurous sulphate there is present. If mercuric sulphate be treated with the acid, no sulphur dioxide is evolved. In fact, if a prepared mixture of mercurous and mercuric sulphates in varying proportions, or any of the crystalline compounds obtained in any of the experiments cited, except that produced by the boiling acid, be treated with fresh acid and heated until the gas evolved ceases to bleach a weak potassium permanganate solution, the crystalline residue then obtained is mercuric sulphate.

Although the relative yield of sulphur dioxide is less, the treatment of mercury with concentrated sulphuric acid has been recommended for class-room experiment on account of neatness and freedom from black residue, which in the case of copper is oftentimes unintelligible to the ordinary student. A slight objection may be urged against this use on account of the volatility of the metallic mercury at the temperature of reaction. Faraday has shown that in spite of its high boiling-point, mercury volatilises at the ordinary temperatures. One of us (Baskerville) has had this fact to contend with in several cases where this method of evolving sulphur dioxide was made use of in quantitative work.

In the experiments given all materials used were pure. The concentrated sulphuric acid was of 1.84 sp. gr. The mercury was purified according to Lothar Meyer's method (*Zeit. Anal. Chem.*, ii., 241), by allowing the distilled metal to flow in a thin stream first through a column of dilute nitric acid, then dilute sulphuric, to dissolve out all lead and tin, finally washed and dried. All apparatus was most carefully cleansed, and when necessary moisture-free and proved to be air-tight.—*Journal of the American Chemical Society*, vol. xix., No. 11.

THE ANALYSIS OF BEARING-METAL ALLOYS, WITH A NEW VOLUMETRIC METHOD FOR DETERMINING COPPER.*

By W. E. GARRIGUES.

JUDGING by the number of articles that continue to appear from year to year in chemical journals, elaborating more or less complete schemes for the analysis of these alloys, the subject is still a timely one for discussion. There is, furthermore, small likelihood that this apparent interest will lag, in the near future at least, owing to the evident fact—and it is one that must not be lost sight of in passing judgment on the merit of such a combination of methods for separation and determination as the present paper deals with—that we are not considering a material capable of being handled like steel, where accurate methods have been found for ascertaining the quantity of almost every element present, without the necessity of first removing three or four others from solution.

In the light of our present knowledge of the chemical deportment of the metals of the hydrogen sulphide and ammonium sulphide groups, all methods which even approach accuracy must of necessity be comparatively tedious. Not alone is it essential in almost every case to free a solution from interfering elements to reach the one sought, but in very many instances it is all but impossible to get the former out of the way without sustaining loss

* Read before the Chemical Section of the Engineers Society of Western Pennsylvania. From the *Journal of the American Chemical Society*, vol. xix., No. 12, December, 1897.

of the latter. Add to this the manipulative difficulties, such as the washing of slimy precipitates that persist in passing the filter medium, and those that in coming down occlude non-volatile salts used as reagents, or sustain loss in ignition through reduction and volatilisation—there is certainly sufficient difficulty remaining to interest many of us for years to come.

The present paper is not written so much with the object of presenting innovations that conduce to speed as to discuss some of the inaccuracies in very commonly used processes, and to considering means for overcoming them, even though at times additional labour may be involved. Of late years the art of successfully working scrap has reached such perfection, that it is probably not an error to assume that the greater part of the metal in alloy bearings, now in use, was at one time doing duty along a varied line reaching from wash-boiler bottoms to tea packages. The inevitable result is that the subject has grown still more intricate, from the analyst's point of view, the range of elements existing in small percentages being greater.

The alloys considered are bronze, brass, and white metal, composed of copper, tin, antimony, lead, zinc, iron, phosphorus, and arsenic.

The advantage of a qualitative analysis, previous to beginning the analysis proper, has often been dwelt upon, and there is but little doubt that it almost invariably repays the trouble tenfold. In the scheme to be outlined here, it is particularly desired to know beforehand whether antimony, iron, zinc, and arsenic are absent; for this purpose we have found the following procedure easy and satisfactory. The sample is oxidised with nitric acid, and evaporated dry, boiled with a little dilute nitric acid, the residue filtered out and washed.

Antimony.—Ignite a portion of the residue intensely, boil with strong hydrochloric acid, and dilute with an equal volume of water. Filter and add considerable water, when a white precipitate indicates antimony. Confirm by orange hydrogen sulphide precipitate.

Arsenic and Iron.—The remaining portion of the residue is warmed with a little caustic soda solution, and dissolved by the further addition of alkali sulphide and heating. The black residue is removed by filtration, and treated for iron by any suitable method. In the alkaline filtrate arsenic is precipitated with ammonia and magnesia mixture, the precipitate filtered out and dissolved in hydrochloric acid, which solution is boiled with sulphurous acid, and then saturated with hydrogen sulphide, when the arsenic, if present, is obtained as sulphide.

Zinc.—The filtrate from the nitric acid insoluble residue is saturated with hydrogen sulphide, the filtrate mixed with one-tenth its volume of strong hydrochloric acid, and potassium ferrocyanide added, when zinc is indicated by a white precipitate of the ferrocyanide.

Quite a large amount of the sample should be used, as otherwise antimony may all pass into the nitric acid filtrate, and thus escape detection. The ignited stannic oxide is entirely insoluble in the boiling hydrochloric acid, while sufficient antimony dissolves to give ample reactions. The large amount of hydrochloric acid is added previous to testing for zinc, to prevent the co-precipitation of any lead that may have escaped coming down as a result of the hydrogen sulphide treatment.

Coming now to the quantitative part of our subject, we have to consider first

The Determination of Single Elements.

Zinc, when present in fairly large quantity, and an absolutely correct determination is not required, can be very conveniently measured by titration with standard potassium ferrocyanide, but in smaller amounts a gravimetric method is preferable. For the details of the process, see Stone (*Journ. American Chem. Soc.*, xvii., p. 413). The end-point is marked by an immediate green colour with cobalt nitrate.

Stone directs that the volume of standard liquor necessary to give the end reaction be determined by a blank experiment, and that this amount be subtracted from each titration. This statement we have been entirely unable to verify, as witness the following set of titrations of varying amounts of the same solution of zinc chloride:—

Zinc chloride. c.c.	Potassium ferrocyanide. c.c.	Total per cent.	Blank per cent.
0	1.5	—	—
5	6.0	30.0	22.5
10	11.8	29.5	25.7
12.5	14.6	29.2	26.2
15	17.5	29.15	26.6
25	29.2	29.2	27.7

Total per cent column is figured on the assumption that the highest titration is per cent zinc, the lower titrations then being multiplied to equal the same volume of zinc chloride used—no blank being subtracted. Figures in blank per cent column are obtained in the same manner, except that the blank is in each case subtracted. It will be noticed that the figures in the latter column are anything but uniform among themselves. To get the best results, the standard solution should be titrated against a known zinc solution of the same strength as the sample under examination. In applying the process absence of copper and iron is essential.

When the conditions of precipitation are fully understood, one of the neatest and most accurate analytical processes is the determination of zinc as phosphate. All metals, inclusive of alkalis, should be absent.

The solution should be exactly neutralised with hydrochloric acid or ammonia, as the case may be, using methyl orange as indicator, unless the liquid contains acetic acid, in which case no further inconvenience results, except that litmus is substituted for methyl orange. A very large excess of ammonium phosphate is to be avoided, as otherwise the precipitate is re-dissolved to some extent. This part of the process is, however, not the delicate matter it might seem from the statement, as will be shown later. The most satisfactory mode of procedure is to add first, say, one grm. of the phosphate, which will not hold up the smallest traces of zinc, and if a large precipitate comes down to continue the addition. Hydrogen sulphide should give no trace of precipitate in the filtrate. It is highly advisable to work only with ammonium salts from the beginning, sodium salts being less good, and potassium salts entirely prohibited, as they are carried down with the precipitate by occlusion in astonishing amounts.

The ammonium phosphate is added to the warm solution of the zinc, which is then further warmed, until the precipitate is entirely free from any flocks, and the solution has completely cleared. Filtering on a Gooch crucible, washing with water and then alcohol, drying at 100° C., and weighing as zinc ammonium phosphate is recommended. Using an ordinary filter paper and igniting seems to cause an unavoidable loss through reduction, no matter what care be exercised in separating the filter. It may, however, be ignited with perfect safety on a Gooch, even though a disc of paper be used as the filtering medium. In the latter case the paper should be turned above the precipitate toward the mouth of the crucible.

The details of the method, with sodium phosphate as precipitant, appear in the last edition of "Crookes's Select Methods." The original author's statement that the ignition gives low results, owing to volatility of the zinc pyrophosphate, cannot be admitted; in fact our excellent results obtained by ignition on a Gooch prove that such is not the case. The same applies to his claim that the precipitation takes place in an acid solution.

Though a difference of opinion as to what constitutes an acid solution may in some cases exist, it certainly does not seem reasonable to apply the term to a liquid smelling strongly of ammonia. The reactions occurring are probably expressed by the equations:—

- a. $\text{ZnCl}_2 + (\text{NH}_4)_2\text{HPO}_4 = \text{ZnHPO}_4 + 2\text{NH}_4\text{Cl}$.
b. $\text{ZnHPO}_4 + (\text{NH}_4)_2\text{HPO}_4 = \text{ZnNH}_4\text{PO}_4 + 2\text{NH}_4\text{H}_2\text{PO}_4$.

It is even likely that these two stages are entirely separate, since in a cold solution the precipitate is at first as flocculent as an alumina precipitate, while on warming it becomes as dense as barium sulphate. If only the amount of ammonium phosphate called for by the first equation is added, the precipitate remains flocculent, despite continued heating.

The double equation requires nearly five parts ammonium phosphate for one part zinc, and we have successfully thrown down two-tenths grm. zinc with one grm. of the phosphate, obtaining it all as the ammonium salt. The same quantity was also perfectly precipitated with five grms. of ammonium phosphate, but eight grms. left a little zinc in solution. Similarly, 0.008 grm. was completely thrown down with 0.05 and 3.00 grms. respectively, while five grms. caused some re-solution.

Returning to the question of acidity, it will be seen from the equation that monoammonium phosphate remains as a product of the reaction, but the excess of the diammonium phosphate, always used in practice, decomposes on heating to monoammonium phosphate and free ammonia, thus determining the alkalinity of the liquid. The precipitate is soluble with great facility in any kind of acid, or in excess of ammonia. Too much cannot be said in favour of the process for accurate work.

Copper is determined in smaller quantities by the iodide method, and in larger quantities by the new process described below. The latest literature added to the iodide method is Low's paper (*Journ. American Chem. Soc.*, xviii., p. 458). He introduces two modifications: Oxidation of any arsenic present, with potassium chlorate, and neutralisation of the excess of nitric acid with zinc acetate.

There is no doubt, as Low states, that the end point is better, as a result of the use of zinc acetate, than when the sodium salt is employed; the oxidation with chlorate is also a great step in advance for the process. It is, however, a mistake to use any acetate, as the end-point is then at best not clear, though with practice it can be distinguished.

Some time ago the writer had the honour to present to this Section a paper on the iodide method for copper, in which it was proposed to avoid the inaccuracy introduced by the presence of arsenic, by separating the copper from it with glucose in the well-known manner. The object was to retain the clear end-point when the titration is conducted in a very slightly acid sulphuric acid solution. Since that time the following, relative to the subject, has been ascertained:—

When copper and arsenic, either as metals or sulphides, as obtained in the course of analysis, are dissolved in nitric acid, there is very apt to result a solution of the arsenic, both as arsenic and arsenious acid. If now this solution be boiled with chlorate in a fairly concentrated condition, all the arsenic is oxidised to the higher form. Arsenic acid in strongly mineral acid solution, mixed with an iodide, is reduced to arsenious acid with liberation of iodine, causing the result for copper to appear high. In a solution which is, however, only faintly acid from a mineral acid, this reaction does not take place; in fact free iodine will then even oxidise arsenious acid to some extent, but far less so than in acetic solution. In the presence of little acid and arsenious acid, the result for copper would therefore appear low, since the iodine liberated by the copper would be in part consumed by the arsenic, instead of being measured by the titrating liquid. If the copper solution be only faintly acid, and the arsenic all present as arsenic acid, an accurate result for copper is obtained in sulphuric acid solution, and the end point with starch is vastly superior than if either zinc or sodium acetate has been used.

The thiosulphate solution should be standardised on approximately the amount of copper found in the assay, conveniently obtained by measuring out a standard copper

sulphate solution, if the best results are to be gotten. In the writer's experience a little more than the theoretical amount of iodine is liberated, and this not in absolutely uniform ratio. For small amounts of copper it is advisable to allow the solution to stand a given time, say ten minutes, between the addition of the iodide and the titration, otherwise the end-point will come and go in a rather unsatisfactory manner.

While the method is good, very much to be preferred to the cyanide titration of Parks and Mohr, after an extended experience with it, we must certainly dissent from Low's claim of superiority for it as against the electrolytic assay.

(To be continued).

NOTICES OF BOOKS.

Notes on Lead and Copper Smelting and Copper Converting. By HIRAM W. HIXON. New York and London: The Scientific Publishing Co. 1897. Pp. 116.

In the preface to this excellent work the author informs us that it was not his intention to write a treatise of the subject, but rather to give the benefit of his practical experience to others engaged in similar work: in this we think he has entirely succeeded.

The book is divided into seventeen chapters, and contains twenty-four illustrations, besides eighteen plates.

In Chapter I. the operation of copper matte smelting is gone into in many of its details, both with regard to the charge, the composition of the slag, wind required, &c. With regard to the relative merits of the Roots and the Baker blowers, Mr. Hixon declares entirely in favour of the former, as there is much less leakage from it than from the Baker blower, and the cost of working must necessarily be less.

The first attempt at recovering silver from the dump of accumulated speiss at the Arkansas Valley Works was not a success; the lumps of speiss were too big for the crusher, and considerable damage was done to the machinery; but as the yield of silver was so high, viz., 5 per cent, repairs and improvements were made and the work resumed. Chapter II., in a few sentences, describes the methods in use for extracting gold and silver from matte.

After a chapter on furnace charges, we come to types of furnaces and the differences necessary in them, for the varying quality and kinds of matter to be smelted or roasted. Spouts, settlers, and jackets are discussed in Chap. V., and in the next chapter the methods of blowing-in and barring-down furnaces are given. The handling of blast-furnace slag is always an important point of the management, and the author considers that there are now four methods in use for lead furnaces, and two for copper furnaces, all of which possess great merit. These are considered *seriatim* in Chap. VII.

The next two chapters concern the design of lead blast-furnaces, and the loss attaching to them.

Many improvements in roasting furnaces have been introduced during the past five years, especially with regard to roasting concentrates; the author has designed a roasting furnace that can be coupled direct on to a reverberatory, and the heat from the smelting thus used for roasting, as well as for the conversion of the matte. This is described in Chap. X., with illustrations. In Anaconda the experiment was tried of smelting raw concentrates with hot-blast, but the results were not satisfactory, and, as with copper converting at the same place, it was found that the expense of the linings was too great. Various linings are discussed in Chap. XII., and we next come to the operation of blowing a converter charge; it is a delicate operation, judged to a great extent by the colour of the flame at the nose of the converter, and necessarily demands great skill, only to be obtained by practice. The formation of copper oxide does not occur to any great

extent so long as any iron is present in the matte; but as soon as the iron is all oxidised, and has gone into the slag, the copper commences to oxidise rapidly, and this is the principal thing to guard against.

Chapters XIV. and XV. are devoted to the design and lining of converters; they are in general appearance just like Bessemer steel-converters.

In the Appendix we have specifications of buildings and machinery for copper converter plant, accompanied by some excellent plans and drawing.

The index is good and comprehensive, and the whole printed on very good paper in the usual first-class American style.

Chemistry for Photographers. By CHAS. F. TOWNSEND, F.C.S., F.R.P.S. Illustrated, pp. 158. London: Dawbarn and Ward, Lim.

THERE is perhaps no science which has made such remarkable—and to some extent startling—strides of late years as photography, and for this very reason books of this character are essential. Kodacs, cinematoscopes, and such like, have raised a growing desire among the public to take photographs for themselves; but unfortunately they are too prone to go on the "You press the button and we do the rest" principle, and therefore have not much knowledge of what they are really doing.

Photography is largely chemistry, and, as the author points out, "the first essentials to anyone studying chemistry are accuracy and cleanliness." This book differs from most books on the same subject, in that it pays much more attention to the chemical side of the subject than is usual. The reagents used, such as "hypo," silver salts, oxidising and reducing agents, are explained, and their chemical action and formulae described, apart from the rule-of-thumb photographic use of the reagents. This, however, is not all; we find plenty of purely technical matter, with all the latest methods and improvements in printing, toning, &c.

One of the most valuable parts of this volume is the "Cyclopædic Index," in which not only every article mentioned in the text is indexed alphabetically, but the index is divided into eight columns, setting forth the name, the reference pages, other names, symbol, molecular weight, solubility in 100 parts of cold water, boiling water, cold alcohol. For instance, we find in the above order:—Sodium thiosulphate; 31, 46, 54, 77, 78, 121; hyposulphite of soda, "hypo"; $\text{Na}_2\text{S}_2\text{O}_3 + 5\text{H}_2\text{O}$; 248; 100; very soluble; insoluble.

The book is handy in form and printed in clear type, and will, we feel sure, meet with the success it deserves.

The Production of Precious Stones in the United States in 1896. By GEORGE F. KUNZ. Washington: Government Printing Office, 1897. Pp. 39.

THE most important events in connection with precious stones during the year 1896 are stated to be the presentation of some interesting conclusions as to the origin of certain diamonds found in Wisconsin by Prof. Hobbs; the publication of Prof. Carvill Lewis's conclusions as to the genesis of the diamond; the finding of sapphires in Montana, equal in colour to, though smaller than, the best Ceylon gems; the continued finding of tourmalines, beryls, and turquoises; and the visit of M. Moissan to the United States to deliver his lecture on artificial diamonds.

The above-mentioned subjects are all given at considerable length in these pages, together with a good deal of other interesting matter, with regard to the finding of precious stones in other parts of the world.

A list of the values of the different gems and precious stones found in the United States during the year 1896 is given at the end, and we note that the turquoise comes first, at 40,000 dollars, followed by sapphire and gold quartz at 10,000 dollars each, and so on down to

dumortierite in quartz at only 50 dollars, the grand total for thirty-eight separate kinds being 97,850 dollars.

The Commercial Uses of Coal-Gas. By THOS. FLETCHER, F.C.S. London, Warrington, and Manchester: Fletcher, Russell, and Co., Lim. Pp. 104.

THE name of Fletcher in connection with gas-stoves is so well known—throughout certainly the English-speaking world—that many lines are not required to describe the handy little book now before us. It must not be thought that this is anything like the well-known laboratory catalogues,—far from it; it is devoted to the economical consumption of coal-gas in its widest sense, as applied not only to heating rooms and cooking, but to the arts and manufactures in every direction; to heating bookbinders' tools and printers' rollers; to the prevention of burglary and the hardening of steel; to bacon drying and smoke abatement; and other objects too many to specify. Mr. Fletcher has done much for the improvement of gas consumption, and deserves the thanks of many thousands of persons.

Handbook to the Workmen's Compensation Act, 1897. By M. ROBERTS-JONES, Barrister-at-Law. London: 82, Fleet Street. Cardiff: Western Mail, Lim. Third Edition. Pp. 86o. 1897.

LARGE employers of labour will find this handbook of great value, by enabling them to ascertain more clearly how the Workmen's Compensation Act works in practice. It is beyond our province to discuss in these columns the wisdom or fairness of the Act; it is enough that it is law, and must be conformed to. Ignorance being no excuse in the eye of the law, a work of this character is of great assistance to those who, through no fault of their own, may become liable to pay damages in respect of accidents. A number of cases are cited which have already come before the courts, and schemes are given whereby the masters and men can arrange a basis of compensation by mutual consent; such schemes, of course, being subject to the approval of the Registrar of Friendly Societies.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxv., No. 26, December 27, 1897.

Constitution of Phthalic Green.—A. Haller and A. Guyot.—The authors have attempted to attribute a formula to phthalic green which would be in harmony with that of diphenylanthrone. Taking analogy into consideration, they think that the green should come from the chlorhydrate of hexamethyltriimidodiphenylanthrone, but this does not altogether fit in. Eventually they decide to adopt the formula suggested to them by M. Rosenstiehl,

$$\begin{array}{c} \text{Cl} \\ | \\ \text{C}_6\text{H}_4 \text{---} \text{C} \begin{array}{l} \text{---} \text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \\ \text{---} \text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \end{array} \\ | \\ \text{CO} \text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \end{array}$$
 which makes of their molecule a malachite green substituted in ortho by $\text{COC}_6\text{H}_4\text{N}(\text{CH}_3)_2$.

Preparation of Alloys of Glucinum. Alloys of Glucinum and Copper.—P. Lebeau.—Alloys of glucinum have been prepared by reducing the oxide of glucinum in the presence of another oxide or a metal. When a mixture of oxide of copper, oxide of glucinum, and carbon is heated in the electric furnace, a well-melted ingot is obtained being an alloy of these two metals. The proportions used in two experiments were—

	I.	II.
Glucina	25 grms.	25 grms.
Oxide of copper ..	50 "	100 "
Carbon	10 "	25 "

In the first case, the resulting ingot weighed 45 grms.; and in the second case, 142 grms. Alloys containing about 10 per cent of glucinum are of a pale yellow colour, almost white; those containing 5 per cent are more yellow; they are easily worked and take a good polish.

Impurities in Aluminium and its Alloys.—Ed. de Facqs.—The insoluble residue left after attacking aluminium with hydrochloric acid was found to consist of oxides of iron and aluminium, silicon, and traces of copper.

On a Double Carbonate of Soda and Protoxide of Chromium.—G. Baugé.—When we add a solution of carbonate of sodium in boiled water to well-washed and still moist chromous acetate, we notice that the acetate immediately goes into solution, and that a reddish brown body is precipitated. This precipitation must be conducted in a current of carbonic acid quite free from oxygen. Chromous sodic carbonate forms two hydrates—one taking ten molecules of water, and the other only one. The former, when examined under the microscope, appears in the form of little reddish brown lozenges—either isolated, or grouped together like the leaves of a book. It is very efflorescent, and is an energetic reducing agent; it decomposes water below 100°, with the disengagement of hydrogen; it is soluble in cold water, but its solubility, however, decreases on keeping. The salt containing one molecule of water is a yellow powder, which changes to brown when heated *in vacuo* or in a current of hydrogen; it regains its yellow colour on cooling. It decomposes at about 300° into green sesquioxide and carbonate of sodium. Heated in the air, it quickly oxidises, forming chromates of sodium. Cold boiled water gradually transforms this salt into the one containing ten molecules of water. The formulae for these two salts are— $\text{CO}_3\text{CrCO}_3\text{Na}_2 \cdot 10\text{H}_2\text{O}$ and $\text{CO}_3\text{CrCO}_3\text{Na}_2 \cdot \text{H}_2\text{O}$.

Atomic Weight of Cerium.—MM. Wyruboff and Verneuil.—A continuation of the discussion with M. Boudouard, in which the authors express the opinion that the differences between them and the last-named gentleman rests on a misunderstanding.

The Aromatic Diurethanes and Piperazine.—P. Cazeneuve and M. Moreau.—Piperazine gives urethanes of the form $\text{CO} \begin{array}{c} \text{N} \begin{array}{c} \text{C}_2\text{H}_4 \\ \text{C}_2\text{H}_4 \end{array} \text{N} \\ \text{OR} \quad \text{RO} \end{array} \text{CO}$; R being an aromatic radical. These are veritable diurethanes formed in virtue of the repetition of the amine group in the molecule.

Use of Carbide of Calcium for the Preparation of Absolute Alcohol.—P. Yvon.—Will be inserted in full.

On α -Acetylfurfurane and its presence in Stockholm Tar.—L. Bouveault.— α -Acetylfurfurane, whether produced synthetically or extracted from tar, is identical both in crystalline form and chemical properties; they each form a crystalline oxime melting at 104°.

Behaviour of a Mixture of Pyridine with Propionic, Acetic, and Formic Acids during Distillation.—G. André.—There is evident combination between these acids and pyridine, for at the moment of mixing there is a considerable disengagement of heat.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xvii.-xviii., Nos. 20-21.

Dissociation Spectra of Melted Salts. Metalloids: Chlorine, Bromine, Iodine.—A. de Gramont.—Already inserted in full.

Reduction of Molybdic Anhydride by Hydrogen, and the Preparation of Molybdenum.—M. Guichard.—In view of the contradictory results obtained by different chemists, and to decide which oxides among Mo_2O_3 ,

Mo_2O_3 , MoO_3 , Mo_2O_3 can be obtained by the action of hydrogen of MoO_3 , the author took up the research methodically. M. Moissan has shown that the three most important factors in such an operation are the rate of flow of the gas, the temperature, and the time. As a rule, the higher oxides pass through intermediate stages before being reduced, but each oxide has a temperature below which reduction does not take place. Hydrogen, washed with chromic acid and dried with potash, was passed over red-hot copper, dried again, and passed through a U tube containing the oxide to be reduced, the U-tube being kept in a bath of melted tin. On slowly heating molybdic anhydride in hydrogen, the colour changes as the temperature rises to yellow, grey, bluish grey, and violet-brown, until eventually, when a red heat is reached, it is entirely of a metallic grey colour. The temperatures at the different colours have been found experimentally not to exceed 600°, and the author has also proved that there are only two anhydrous oxides, MoO_3 and Mo_2O_3 , the others previously mentioned not being definite compounds.

Modification of Friedel's and Crafts's Method giving a Higher Return in Syntheses made by the Help of Chloride of Aluminium.—A. Verley.—A long paper not suitable for abstraction.

Action of Nitrate, Sulphate, Chlorate, and Phosphate of Ammonia on *Aspillergerus Niger*.—C. Tanret.—The author is of the opinion that *aspillergerus* and the higher plants ought equally to be able to get rid of the mineral acids which are of no use to them, and that the acid excretion which has been observed in their radicles should be due to these acids, either in the free state or in combination with organic matters.

No. 22.

Some New Experiments on the Liquefaction of Fluorine.—MM. Moissan and J. Dewar.—Already inserted in full.

Chromosulphochromic Acids. Alkaline Chromosulphochromates.—A. Recoura.—The author describes certain compounds analogous to those formed by the combination of the green sulphate of chromium with sulphuric acid, but in this case with chromic acid and the chromates. The existence of these compounds can easily be shown by adding a solution containing one molecule of $\text{Cr}_2\text{S}_3\text{SO}_4$ to a solution containing one molecule of chromic acid or chromate of potash; the combination takes place immediately, and that the sulphuric and chromic acids are both assimilated is proved by the fact that neither chloride of barium nor nitrate of silver will give a precipitate. The same combination of the green sulphate can be shown with two molecules of chromic acid or chromate of potassium; and the chromosulpho-dichromate of potassium, when dried at 150°, is, like the monochromate, insoluble in water. In a similar manner chromosulpho-trichromic acid can be obtained, but the combination stops here, for if four molecules be added a precipitate is obtained by nitrate of silver, when all the other conditions are exactly the same as before.

Action of Oxygen on Dissolved Formic Aldehyde.—Marcel Delépine.—Although oxygen has no action on an aqueous solution of formic aldehyde at ordinary temperatures, if it is warmed up to 200° the whole of the oxygen enclosed in the tube is absorbed. The presence of potash in the cold, or even at 100°, will not cause the oxidation of dissolved formic aldehyde, but in the presence of spongy platinum complete oxidation—a veritable combustion—takes place at the ordinary temperature.

Action of Chloride of Ethylalcohol on the Aromatic Hydrocarbons.—L. Bouveault.—A number of new organic compounds are described, but the paper is hardly suitable for abstraction.

Glyoxylic Acids and Aldehyds derived from the Phenol Ethers.—L. Bouveault.—Not suitable for abstraction.

On Phenol-Glyoxylic Acids.—L. Bouveault.—The simple phenols give no reaction with chloride of ethyl-oxalyl and chloride of aluminium, neither do the radical-acid ethers of the phenols; but the best results have been obtained with the ethers formed by picric acid. Several of these compounds are described.

On some Derivatives of Guaiacol.—L. Bouveault.—The unsuccessful attempts made by the author to effect the synthesis of vanilline caused him to prepare several ether derivatives of guaiacol, which are here described.

Volumetric Estimation of Combined Sulphuric Acid.—Felix Marboutin and Marcel Molinié.—The authors have undertaken to determine the corrections necessary, in the Windisch method, of estimating sulphuric acid volumetrically, on account of the unavoidable non-equivalence of the two solutions used, viz., chloride of barium and chromate of potassium. In their experiments they used N/50 solutions, but the chromate was of a little higher value than the barium; the figures they obtained were compared with those of the gravimetric method, and a large table given enables us to compare the relative values of the two volumetric methods, and it appears that the new modified method is superior in exactitude to that of Windisch.

Volumetric Estimation of Combined Sulphuric Acid.—Felix Marboutin.—To estimate the sulphates in potable waters, 100 c.c. of the water is acidulated with hydrochloric acid, and boiled to drive off the carbonic acid; the temperature is then slightly lowered, and 30 c.c. of chloride of barium is added, drop by drop; then let stand at 40° to settle; it is then neutralised with a few drops of ammonia, and 30 c.c. of chromate of potassium added; the liquid is made up to 300 c.c. after cooling. To 100 c.c. of the clear liquid is added 2 c.c. of 25 per cent sulphuric acid and 5 c.c. of arsenious acid, it is then warmed and shaken till the colour disappears. After neutralising with carbonate of potassium, titrated iodine is added until starch is coloured. This method has the advantage of requiring no filtration or washings, and it takes into account any impurities in the reagents, and they need not, except the iodide, be very carefully titrated beforehand.

MISCELLANEOUS.

Nottingham University College.—The Retirement of Dr. Clowes.—An interesting little ceremony took place last evening at the Nottingham University College, when a number of the past and present chemical students, together with the chemical staff of the college, assembled for the purpose of making a presentation to Dr. F. Clowes. Until recently Dr. Clowes occupied the chair of chemistry at the University College, which he has held since the opening of the institution 16 years ago. He recently resigned the position in order to assume the office of chemical adviser and chief chemist to the London County Council. The Rev. Principal Symes presided, and among those also present were Prof. Granger, Prof. Robinson, Dr. Clowes, Dr. Kipping, and Dr. Sudborough. The presentation consisted of a handsome silver kettle for the tea-table, and ornamental silver candlesticks. The Rev. Principal Symes alluded to the ability and patience which Dr. Clowes had displayed during the sixteen years he had been engaged at the college. In earlier years the institution had to pass through a period of exceptional difficulties, and they owed to Dr. Clowes a deep debt of gratitude for his tact and courtesy during those days. (Applause). Those were very critical times, when the very existence of the college was at stake. The governing body of the institution had just testified to their sense of appreciation of the work which had been accomplished by Dr. Clowes, by bestowing upon him the title of *emeritus professor*.

(Applause). In conclusion, Professor Symes said that they all wished for Dr. Clowes a long and useful career in his new sphere. They were sure that if it was lengthy it would be useful. (Applause). Dr. Sudborough and representatives of the past and present chemical students also spoke. Dr. Clowes acknowledged with deep gratitude the handsome presentation, which, valuable in itself, would serve to him as an invaluable memento of kindly feelings entertained towards him by those with whom he had been associated as fellow worker for sixteen years. His own chemical department had been more especially dear to him, and his relationship towards staff and students in that department had always been one of personal and individual friendship. This kindly and generous reception of his work had rendered his duties a real source of enjoyment to him, and would always be remembered by him with pleasure. It resulted in his meeting friends, who had been associated with him as fellow teachers or pupils in all parts of the country, and, indeed, of the world. (Applause). After nearly twenty-five years spent in active tuition, he was not sorry to assume advisory and consultative duties only. In his new sphere, acting as chemical adviser and chief chemist to the London County Council, he had the direction of a staff of over forty assistants, most of whom were qualified chemists, and he hoped that he might, therefore, be able to find useful employment for some of those who had received their chemical training in the Nottingham College. The position involved advice and investigation in connection with chemical problems of the most varied description, but it included the constant examination of the metropolitan gas and water supplies, the provision of the best means for disposal of the London sewage, and the maintenance of the purity of the Thames, more especially in its tidal portion. For the performance of these duties twenty gas-testing stations were provided, in which daily tests were carried out and reported to the chief chemist; two chemical laboratories at the sewage outfalls, whence the results of the chemical examination of the sewage effluent into the Thames and of the tidal Thames were periodically reported, and a central laboratory at Charing Cross, in which general chemical analysis and investigation were constantly being carried out. As a result of the past work in this chemical department, the condition of the gas supply to London had been vastly improved, and the Thames had ceased to be a foul offensive stream within the metropolitan reaches, whilst the use of bad mortar by the jerry-builder and the introduction of dangerous petroleum has almost ceased. It is hoped that ere long the fish, which had now made their appearance again in the tidal portions of the river, would be able to pass through London to the upper stream. In conclusion, Dr. Clowes mentioned the general scope of his new duties, for the reason that many of his friends in Nottingham were asking for the information. They were duties which, connected as they were with a population of nearly five millions, would prove engrossing and important, but he was sure, that while engaged in the large sphere, memory would frequently transport him to the period of happy work carried out in Nottingham. (Applause).

Institute of Chemistry January (1898) Examinations.—Names of Candidates who passed the Practical Examination for the Associateship (under Regulations in force prior to October 1st, 1893).—William Alfred Caldwell, Glasgow and West of Scotland Technical College; Alfred Campion, jun., Finsbury Technical College, London; George P. Darnell-Smith, B.Sc. (Lond.), University College, London, and under Professor J. Wertheimer, F.I.C., Merchant Venturers' Technical College, Bristol; Albert Edward Thomas, registered student under Professor J. Wertheimer at the Merchant Venturers' Technical College, Bristol; Alfred John Turner, Finsbury Technical College, London.

Final Examination for the Associateship (New Regulations).—In Section "A" (General Inorganic Chemistry):

Alexander Hugh Dewar, Glasgow and West of Scotland Technical College. In Section "B" (the Chemistry of Metals and Alloys): Albert Lucas Entwistle, Owens College, Manchester, and registered student under T. J. Hutchinson, Esq., F.I.C. In Section "D" (Organic Chemistry): Bertram William John Warren, Mason College, Birmingham, and Finsbury Technical College, London. In Section "E" (the Chemistry of Water, Food, and Drugs): Edwin James Read, B.A. (Cantab), Cambridge University.

Intermediate Examination (New Regulations).—Norman Parr Booth, Mason College, Birmingham; Charles Adolphus Hackman, King's College, London, and with A. C. Chapman, Esq., F.I.C.; Leonard Myddelton Nash, King's College, London, and with Bertram Blount, Esq., F.I.C.; Roland Cecil Wild, King's College, London, and registered student under C. J. Head, Esq., F.I.C.

The Royal Photographic Society is organising an International Exhibition of Photographic Apparatus and Photographs, which will open at the Crystal Palace on April 27th. In addition to the usual displays of pictures, &c., the leading firms, manufacturers, and dealers will be largely represented. There will also be extensive loan collections, illustrating not only the history of photography, but its enormous scientific and commercial applications, photo-mechanical processes, photographs in colours, photographs by means of the X-rays, and kindred exhibits. The Exhibition, the arrangements for which are in the hands of a joint committee of members of the Society and exhibitors, bids fair to be the largest and most interesting collection dealing with photography which has ever been got together.

The Sanitary Institute.—The Council have accepted an invitation from the Lord Mayor and City Council of Birmingham to hold its Seventeenth Congress and Exhibition in that city in September next.

Nature of the Compounds of Antipyrin with the Aldehyds.—G. Patein. — Antipyrin combines with aldehyds; the union being effected by carbon, never by nitrogen. In these compounds, which are true derivatives of methane, the atom of nitrogen united to methyl loses the power of combining with chloral and the phenols. Chloral, or trichloric aldehyd can unite with antipyrin only by means of nitrogen, and never by carbon, like the non-substituted aldehyds.—*Comptes Rendus*, vol. cxxv., No. 23.

MEETINGS FOR THE WEEK.

MONDAY, 31st.—Society of Arts, 8. (Cantor Lectures). "Decorative Bookbinding," by Cyril Davenport.

TUESDAY, Feb. 1st.—Royal Institution, 5. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

WEDNESDAY, 2nd.—Society of Arts, 8. "The Cinematograph," by Jules Fuert.

Society of Public Analysts, 8. Annual Meeting to be held at the Chemical Society's Rooms, Burlington House. President's Address. "Copper," "Pure for Analysis," by J. W. Westmoreland. "Note on the Tests for distinguishing Boiled from Unboiled Milk," by H. Leffmann, M.D. Election of Officers and Council.

THURSDAY, 3rd.—Chemical, 8. "On the Dissociation of Platinichloride in Dilute Solution, and the Production of Platinum Monochloride," by E. Sonstadt. "Effect of the Mono-, Di-, and Tri-chloroacetyl Groups on the Rotatory Power of Methyl and Ethylic Glycerates and Tartrates," by Percy Frankland, F.R.S., and Thomas Stewart Patterson, Ph.D. "The Rotation of Ethylic and Methyl Di-monochloroacetyl tartrates," by Percy Frankland, F.R.S., and Andrew Turnbull, Ph.D. "The Volumetric Estimation of Sodium," by H. J. H. Fenton, M.A.

Royal Institution, 5. "The Halogen Group of Elements," by Prof. Dewar, M.A., LL.D.

FRIDAY, 4th.—Royal Institution, 5. "Some New Studies in Cathode and Röntgen Radiations," by A. A. C. Swinton.

SATURDAY, 5th.—Royal Institution, 3. "Cyprus," by Prof. Patrick Geddes, F.R.S.E.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Destruction of Moths.—(Reply to "Moth-eaten").—One of my friends, a physician, tells me that he placed an Astrachan fur cloak already attacked by moths in a tin receptacle, introduced 10 c.c. of formalin (asserted to contain 40 per cent of formic aldehyd), and covered and sealed the tin with care. After a considerable time he opened the tin, when out flew a cloud of moths. My friend is a bacteriologist; he finds that formaldehyd is destructive to many forms of bacteria, but by no means to all; and, so far as his experience has gone, it has but little effect upon animal life such as moths and other household pests.—ELWYN WALLER, New York, U.S.A.

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THE RESEARCH SOCIETY.

IN Appendix F to Part II. of the new edition (1890) of Thomson and Tait's "Treatise on Natural Philosophy" the Kinetic Theory of Gases is stated to be "an established truth of science."

At page 226 of Vol. I. of Lord Kelvin's "Popular Lectures and Addresses" (*Nature* Series) there occur the words—"What we now accept as a surest article of scientific faith—the Kinetic theory of gases."

In §22 of the "Text-book of Physical Chemistry" (1897), by Prof. Clarence L. Speyers, of Rutgers College, the statement is made that "The Kinetic theory is a troublesome thing and is becoming an object of ridicule. It has never directed the chemist to any new discovery or idea, unless it may be Van der Waal's theory, and that would probably have come anyway."

These quotations amply suffice to establish the need that exists for a school of philosophy based upon the *facts* of Nature as distinguished from the mere utterances of Authority.

(In saying this no disrespect is intended to Lord Kelvin and Prof. Tait. They deservedly rank high in the hierarchy of Science. But if Prof. Speyers be correct in his description of the Kinetic theory of gases, it follows that much of the teaching of Thomson and Tait is misleading).

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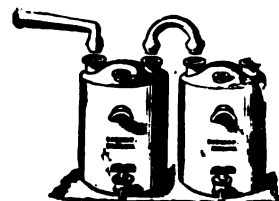
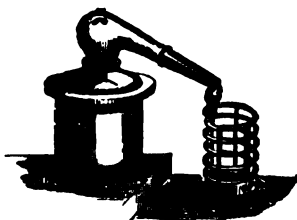
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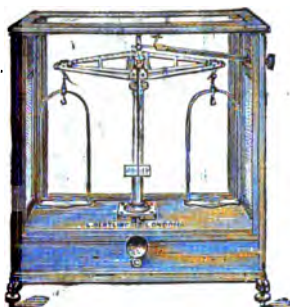
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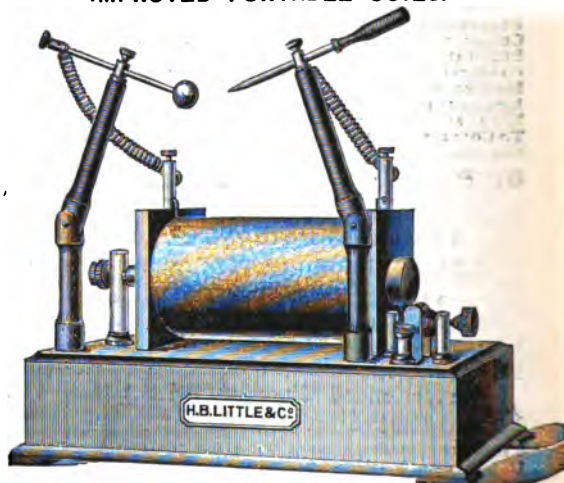
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THE CHEMICAL NEWS.

VOL. LXXVII., No. 1993.

SEPARATIONS FROM CHROMIC ACID.

I. THE SEPARATION OF IRON.

By HARRY BREARLEY.

a. A Reaction between Ferric Salts and Alkaline Chromates.

In a former paper (CHEMICAL NEWS, lxxvi., p. 175) it was pointed out that a customary means of separating iron and chromium, viz., precipitation of the former as basic acetate after oxidising the latter to chromic acid, contained an inherent source of error. This was traced to a reaction between solutions of ferric chloride and alkaline chromates, which it is now proposed to examine more closely.

In another form, or degree, this reaction is already well known, having been observed as early as 1829, and subsequently studied by a number of chemists. In 1862 Storer and Eliot (CHEM. NEWS, vi., p. 12) again observe the reaction and summarise the work of previous observers.

It should, however, be mentioned that the reaction between ferric chloride and potassium chromate occupies quite a secondary place in the preceding researches. The chief aim was to determine the composition of the precipitate formed by adding potassium chromate to a solution of a salt of chromic oxide. Storer and Eliot's title, "On the Chromate of Chromium and Analogous Chromates," is sufficiently expressive of the central interest.

The reaction between solutions of the salts of CrO_3 and Cr_2O_3 was known to Vauquelin, the discoverer of chromium, and twenty years later, guided by the analogy of chromium and iron, Thompson prepared a compound of chromic acid and ferric oxide. This he found to be very basic (Fe_2O_3) $_2$ $\cdot$ Cr_2O_3 , and in the filtrate and wash-waters he thought he found another less basic chromate, (Fe_2O_3) $_2$ $\cdot$ CrO_3 .

A great deal of work was done with an eye to determining whether the chromic acid and chromic oxide compound should be viewed as an oxide, CrO_2 , or as a chromate, $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$. The latter view was supported by analogy with $\text{Fe}_2\text{O}_3 \cdot \text{CrO}_3$, $\text{Al}_2\text{O}_3 \cdot \text{CrO}_3$, and $\text{Mn}_2\text{O}_3 \cdot \text{CrO}_3$, all of which are said to be less stable compounds than $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$; and this even cannot be washed with water without being changed into a more and more basic compound approaching finally to Cr_2O_3 .

It is manifest that these precipitates could be prepared of the required basicity only by mixing concentrated solutions of the two salts. In this respect the conditions differ from the usual iron-chromium separation. The only other points of difference are that in the latter case the iron is largely present as dissolved hydrate, and a reagent (alkaline acetate), whose presence it might be supposed would easily overcome the feeble affinity between the iron and chromium, is added for the express purpose of precipitating the iron. Some such fallacious reasoning would seem, in the first place, to have shielded a chromium-iron separation from suspicion; for later times we see occasionally in chemistry, as elsewhere, that "was grau vor Alter ist, das ist ihm gottlich."

It is too much to say that an error has never been suspected. The considerable abandonment of the method shows that it has, but that the method was fundamentally unsound does not seem to have entered into anybody's reckonings, and later references will show that it is still so far trusted as to beget yearly one or two modifications.

In pointing out the shortcomings of this method it seemed proper to test the matter under such conditions as obtain in practice, and to neglect its relation to the wider questions mentioned above. These conditions are dilute solution, and iron as ferric chloride and as dissolved ferric hydrate.

This arrangement permits the iron to become allied with the chromate in two ways:—1st, with ferric chloride in a manner strictly analogous with the procedure of Storer and others; and 2nd, the ferric chloride having been decomposed, by reacting with the ferric hydrate. The consequent changes are considered separately.

a. Reaction with Ferric Chloride.

The reaction for the production of $\text{Fe}_2\text{O}_3 \cdot \text{CrO}_3$ has been formulated so:—



and, according to Storer, until the requisite amount of chromate has been added the precipitate once formed redissolves,—if the solution is cold. In a hot solution, however, a precipitate forms when only about one-fourteenth part of this chromate is present.

Thus, using 1 gm. of iron as ferric chloride and 50 c.c. of a K_2CrO_4 solution (12½ grms. per litre), in a volume of 1 litre it was found that on boiling a precipitate formed, which, although unfilterable, settled completely after standing several days, and an analysis of the decanted solution showed that the precipitate contained chromium equivalent to 29.2 c.c. of the K_2CrO_4 solution, and iron equal to only 0.6527 gm. With intent to effect a more complete separation of the iron there was added 70, 100, and 120 c.c. of the chromate. The solutions were allowed to cool and settle, although with 70 c.c. and upwards filtration is possible. Table I. shows the relative composition of the precipitates, determined by analysing an aliquot part of the clear supernatant solution.

TABLE I.

Chromate added.	In precipitate.	
	C.c. chromate.	Grm. iron.
50 c.c.	29.2	0.6527
70 "	40.3	0.7848
100 "	44.0	0.7609
120 "	46.3	0.7137

As the two latter ones had been standing two or three days, it seemed likely that the cold solution had redissolved the precipitate. This was found to be the case, and, moreover, it was found that the precipitate redissolves more and more as it stands, and is shaken from day to day; and not the CrO_3 only, as some writers would lead one to infer, but the iron also is dissolved. At first the precipitate seems to become less and less basic, and then the iron and chromium are dissolved proportionately and the composition of the precipitate remains constant; but this composition varies with the amount of K_2CrO_4 originally used as precipitant. These changes, interesting enough in another connection, have, however, very little to do with the present purpose.

b. Reaction with Ferric Hydrate.

An acid solution of ferric chloride, representing 1 gm. of iron, was precipitated from a hot solution, with an equivalent amount of soda carbonate, the chromate added, solution made up to a litre and boiled. The results are shown in—

TABLE II.

Chromate added.	Recovered from filtrate.	In precipitate.
10 c.c.	2.0 c.c.	8.0 c.c.
30 "	16.2 "	13.8 "
50 "	34.2 "	15.8 "
100 "	81.6 "	18.4 "

It is proper to draw attention to the fact that in each of the above cases the supernatant chromic liquid was sepa-

rated by fractional filtration. It is common, however, although more tedious, to filter completely and wash the precipitate. Such a procedure might have shown a more complete separation in some of the cases, but it could hardly have made up the deficiency, because, presuming that all the cohesive liquid was removed, the basic chromates are decomposed only very slowly by washing. In the present case, too, when the washings ceased to be alkaline, the precipitate might also be dissolved (Table I.) as well as decomposed.

B. The Relation of the Reaction to some Analytical Methods.

Our attention was drawn to this matter by attempting to separate iron and chromium by means of soda acetate. This method is usually attributed to Gibbs. It is stated fully in the CHEM. NEWS (xi., 101). It is to be found also in Crookes's "Select Methods," Fresenius's "Quantitative Analysis," "Industries" (1893), and in other publications, so that it has probably been extensively used.

From a former paper (CHEM. NEWS, lxxvi., 175) it appears that the larger the amount of acetate used the greater the percentage separation; but it required impracticable amounts to effect even an approximate separation of the chromate from large amounts of iron. In each of these cases the solution contained 1 per cent or more of acetic acid, which did not appear to be advantageous. Its entire absence, in fact, might be desirable if such a circumstance could accompany the separation of iron as basic acetate at boiling temperatures. At lower temperatures, however, ferric acetate may be precipitated without decomposition, and it might be worth while to observe the separation of iron and chromium in this manner, if more promising and less troublesome means were not at our disposal.

As I have said, the shortcoming of this method has been recognised, and there have been proposed a number of methods in which either the oxidant or the precipitant or both are replaced by others. Thus, looking over former numbers of this journal and some recent issues of other journals, I tabulate the following:—

Oxidised with—	Separated iron with—
I.—Gibbs (CHEM. NEWS, xi., 101)— Chlorine, bromine, or lead peroxide.	Soda acetate or ammonia hydrate.
II.—Sell (CHEM. NEWS, liv., 299)— Hydrogen peroxide.	Potash.
III.—Baubigny (CHEM. NEWS, l., 18)— Nitric acid and potas- sium chlorate.	A very slight excess of soda bicarbonate.
IV.—J. Clarke (Journ. Chem. Soc., 1893, p. 1082)— Soda peroxide.	Peroxide added to alka- linity.
V.—Marchal and Wiernik (Journ. Chem. Soc. Abs., 1893, ii., 49)— Freshly precipitated and well-washed MnO ₂ in neutral liquid.	Boiling.
VI.—Giorgis (Journ. Chem. Soc., Abs., 1893, xi., 554)— Permanganate in slightly alkaline solution.	
VII.—E. Clarke (Journ. Amer. Chem. Soc., 1895, p. 327)— Nitric acid and potas- sium chlorate.	Ammonia.

The following notes are abstracted from the respective papers:—

II. The context leads one to infer that only a small excess of potash was used. The results are shown to be low, and are attributed to part of the chromic hydrate escaping oxidation.

IV. "When applied to liquids containing a good deal of iron the results are generally somewhat low, even when

the iron is precipitated twice in this way." Poleck (CHEM. NEWS, lxi., 285), and Kassner (Journ. Chem. Soc., Abs., 1894, ii., 429) base a like process on the same facts.

V. The authors state that the processes of Wohler (oxidation with Br or Cl and precipitation of Fe with soda hydrate) and Gibbs both fail, even qualitatively, if the amount of chromium is small. The abstractor states that the test analyses, made on rather small quantities, are fairly satisfactory.

VII. "To render the separation complete the precipitate must be re-dissolved and re-precipitated by ammonia."

The following series of experiments help to explain the various opinions, to point out a source of error, and to suggest means of obtaining uniformly accurate separations.

It would be in order to severally examine the efficiency of the oxidants to completely oxidise the Cr₂O₃ to CrO₃ in presence of much iron. Certainly they are not all equally effective; some may even be defective. But apart from this there is a source of error—almost certain error—which previous experience leads us to expect, viz., the formation of basic ferric chromates. These, it might be presumed, would be greater or less according to the nature and amount of alkali used to precipitate the iron. Experiments dealing with this part of the subject are summarised in Table III. Each test contained 1 grm. Fe, 30 c.c. K₂CrO₄ (12½ grms. per litre), and a volume of 1 litre. The alkali used was in each case 2-normal strength.

TABLE III.

Excess of alkali.	Percentage separation.		
	Sod. carb.	Sod. hyd.	Ammon. hyd.
0	54.6	69.3	24.8
10	90.4	100.0	70.4
20	97.5	99.8	77.0
30	98.7	100.0	83.6
50	100.1	—	87.8

The figures seem to indicate that either of the precipitants would give a perfect separation if enough of it was used. Soda hydrate is, however, by far the most practicable, and this it will be remembered is the precipitant recommended in Wohler's method.

The analogy in so many respects between iron, aluminium, chromium, and manganese, leads one to think that an examination of these metals in turn, with respect to their separation from chromic acid by the usual methods, may not be unfruitful.

These observations, along with others on the use of alkaline chromates as precipitants, will form the matter of subsequent papers.

ON THE COLOUR OF AMORPHOUS MERCUROUS IODIDE.

By MAURICE FRANCOIS.

INCIDENTALLY I showed in a preceding article (Journ. de Pharm. et de Chim., [6], Nov. 15, 1897), that a solution of mercurous nitrate transforms mercuric iodide into mercurous iodide of a pure yellow colour; we know, on the other hand, that dilute nitric acid easily dissolves finely divided mercury. In view of these two facts it did not appear to be difficult to devise a method for the preparation of pure mercurous iodide free from both free mercury and mercuric iodide. All that is required is that the mercurous iodide shall be formed in the presence of free nitric acid, and mercurous nitrate in considerable excess. This iodide should be of a very pure yellow colour. To effect this we weigh out 125 grms. of air-dried mercurous nitrate, and dissolve it in two litres of water containing 20 c.c. of

nitric acid. To the clear liquid add, drop by drop, from a burette while constantly stirring, a solution of 50 grms. of iodide of potassium in 100 c.c. of distilled water. The quantity of iodide of potassium necessary to precipitate all the mercury would be theoretically 75 grms.

Each drop as it falls produces a grey stain (local excess of KI), becoming yellow on stirring.

After having run in all the iodide of potassium, the liquid must be vigorously shaken for about fifteen minutes, so as to maintain the closest contact between the precipitate and the mother liquors. The precipitate, at first of a greenish yellow colour, has become much brighter. The whole is then left for twenty-four hours in complete darkness, and shaken up at intervals three or four times. After the lapse of this time, the precipitate is of a very pure bright yellow colour (similar to that of the yellow chromate of lead); it is then washed ten times by decantation, still in the dark, using two litres of water for each washing. The precipitate is finally collected on a filter, drained and dried at 50°, still in the dark.

Any washing with alcohol or ether is quite useless, and would change the character of the substance instead of purifying it. The iodide thus obtained is free from nitrate, mercuric iodide, and free mercury. On analysis it has been found to consist of:—

Found (per cent).	Theory (per cent).
Mercury .. 61·06	Mercury .. 61·16
Iodine .. 38·96	Iodine .. 38·84

Having, on several occasions, had in my possession pure amorphous mercurous iodide, of a pure bright yellow colour, without any tinge of green, I was led to enquire what is the colour of pure mercurous iodide.

The generally accepted opinion is that the colour of amorphous mercurous iodide is a greenish yellow. Boullay even called this iodide the *green iodide of mercury*. M. Raoul Varet (*Comptes Rendus*, vol. cxx., pp. 1054-1114) made a distinction between the yellow mercurous iodide and the green mercurous iodide, and he moreover determined the amount of heat which corresponds to the passage of one of these modifications to the other. The commercial samples of amorphous mercurous iodide certainly show a considerable variation in tint.

For the purpose of getting at the root of the question, I analysed a certain number of samples prepared by different methods, and varying in colour from a pure yellow to a dark green. I now give the results of the analyses, together with the methods of preparation:—

I. Bright yellow mercurous iodide (chrome yellow colour), prepared by the action of iodized potassic iodide on mercurous nitrate (see my previous paper). Found:—Mercury, 61·29 per cent; iodine, 38·51 per cent.

II. Bright yellow mercurous iodide (chrome yellow colour), prepared by the action of iodide of potassium on mercurous nitrate, as I have just described above. Found:—Mercury, 60·96 per cent; iodine, 38·90 per cent.

III. Yellowish green mercurous iodide, prepared by the direct action of iodine on mercury, by following exactly the method described in the Codex. Found:—Mercury, 62·96 per cent; iodine, 36·74 per cent.

IV. Another greenish yellow mercurous iodide, prepared by the direct action of iodine on mercury. Found:—Mercury, 63·90 per cent; iodine, 36·00 per cent.

V. Dark green mercurous iodide, prepared by the action of iodine of potassium on calomel.

Found.—Mercury, 82·46 per cent; iodine, 12·11 per cent; chlorine, 5·35 per cent.

Theoretical for Hg_2I_2 .—Mercury, 61·16 per cent; iodine, 38·84 per cent.

As can be seen from this table, which I have purposely cut as short as possible, if the two first specimens can be regarded as pure, then the three last vary considerably from the theoretical composition.

Further, the difference between the weight of mercury found and the theoretical weight does not indicate the

quantity of free mercury present in the sample. To arrive at this excess, it is necessary to calculate the quantity of this metal required by the iodine found to form mercurous iodide. The difference between the quantity found and the quantity calculated is the free mercury contained in the substance. For the last sample, the weight of mercury, combined with chlorine to form calomel, has also been calculated.

Thus we find that Sample III. contains 5·11 per cent, Sample IV., 7·22 per cent, and Sample V., 33·28 per cent of free mercury.

The presence of an excess of mercury in Samples III., IV., and V. is, however, not astonishing.

In the preparation of mercurous iodide, by the direct action of iodine on mercury, we generally use (Codex) a slight excess of mercury, principally because the product is intended for medicinal purposes, and it is of importance that it should not contain any mercuric iodide. Several causes contribute to a still further increase in the proportion of mercury present. For instance, when the substance is prepared on a large scale, the mass heats and iodine is volatilised in great abundance. During the same operation, light causes the production of mercury and of mercuric iodide, which would be carried off by the subsequent washing with alcohol. Finally, this same washing still further enriches the product in mercury, as I have already shown (*Journ. de Pharm. et de Chim.*, vol. iii., p. 231, 1896). In the preparation by the action of iodide of potassium on calomel, the iodide of potassium is in excess with regard to the first portions of mercurous iodide formed, and we well know that it decomposes this body into mercury and mercuric iodide, which goes into solution.

Thus, mercurous iodide of a greenish yellow or green colour always contains a considerable quantity of free mercury. It seems to me only natural to conclude that it is the presence of free mercury which produces the green colouration, and that the colour of pure amorphous mercurous iodide is a bright yellow.

This opinion is supported by a number of old experiments, and some personal observations. For example:—

I. The green colour of various samples of mercurous iodide is different with each sample, tending sometimes towards the yellow, sometimes towards the grey, and verging sometimes on the black. That is not the behaviour of a pure body with a definite colour.

II. We can change the colour of mercurous iodide progressively from a bright yellow to a green by charging it with free mercury, as we actually do by adding small quantities of iodide of potassium to the yellow iodide suspended in water.

III. We can inversely change the green iodide to the yellow by removing free mercury from it; this can be done by two methods:—

(a). Into two flat-bottomed flasks we place 20 grms. of very green mercurous iodide (what was actually used was obtained by carefully acting on commercial mercurous iodide, with iodide of potassium, and then washing). Into one of these flasks we pour 50 c.c. of water (this flask is intended to serve as a check); into the other we pour 50 c.c. of dilute nitric acid (20 grms. of ordinary nitric acid and 80 grms. of water). These two flasks are kept in the dark, and well shaken every day. After several days we notice that the iodide, in the presence of the nitric acid, has become of a bright yellow colour.

(b). We place two grms. of green mercurous nitrate in a flat-bottomed flask, and as before prepare a check flask; add 100 c.c. of a normal solution of potassium iodide, saturated in the cold with mercuric iodide (this must be kept in the dark and well shaken every day). After a fortnight the mercurous iodide has assumed a very beautiful yellow colour. Evidently, in the first case, the free mercury has been changed into nitrate of mercury by the nitric acid; and in the second case, the mercuric iodide has transformed the free mercury into mercurous iodide. The free mercury disappearing, the iodide returns to its

natural colour, bright yellow. Analysis completely confirms these conclusions.

I cannot finish this article without thanking Professor Jungfleisch for the excellent advice which he has constantly given me, and which has aided me considerably in the course of this work.—*Journ. de Pharm. et de Chim.*, Series 6, vol. vi., No. 12, 1897.

ON THE USE OF CARBIDE OF CALCIUM FOR THE PREPARATION OF ABSOLUTE ALCOHOL.

By M. P. YVON.

WHEN coarsely powdered carbide of calcium is brought into contact with concentrated alcohol (90 to 95 per cent) the carbide is fairly vigorously attacked, and continues to give off acetylene as long as any water remains in the alcohol; when the latter is quite anhydrous, the disengagement of gas ceases.

The use of carbide of calcium, therefore, enables us to determine whether or no an alcohol is anhydrous; it suffices, in fact, to place a few cubic centimetres of the alcohol in a perfectly dry test-tube, and then to drop in a pinch of the coarsely powdered carbide of calcium: if the alcohol is absolute, no gas bubbles whatever can be detected, and even after shaking the liquid remains transparent. If, on the contrary, the alcohol contains traces of water, we can see the little bubbles of gas forming, and on shaking, the mixture becomes cloudy and of a milky white appearance, on account of the formation of hydrate of lime.

To prepare absolute alcohol it suffices to place a quantity of alcohol at 90 or 95 per cent in a flask, with one-quarter its weight of carbide of calcium reduced to a coarse powder. The disengagement of gas, at first very vigorous, soon calms down; the flask is then frequently shaken up during two or three hours, and then left alone for twelve hours. It is then best to make quite certain that shaking no longer gives rise to any disengagement of gas; in the contrary case we prolong the agitation and the contact of the alcohol with the carbide, if necessary adding a little more of the latter; we then transfer the mixture to a distilling apparatus, and proceed with the separation of the alcohol, though putting on one side the first portions collected, as they hold a small quantity of acetylene in solution. It is advisable to keep the first portions far away from any flame, as they consist of a mixture of alcohol and acetylene. The alcohol afterwards collected is anhydrous, if the operation has been properly conducted.

It is preferable, however, to collect all the alcohol in the same receptacle, and then shake it up with a small quantity of dried sulphate of copper, which takes up all the acetylene held in solution. We can then proceed with a second distillation without separating the acetylides of copper which is formed.

Absolute alcohol prepared by this method is not precipitated by alcoholate of baryta; carbide of calcium is therefore as sensitive a reagent as this latter, and enables us to obtain absolute alcohol by one distillation, or two at the most, by starting with alcohol at 90 or 95 per cent.—*Comptes Rendus*, cxv., No. 26.

Physical Society. — Mr. Thomas H. Blakesley has resigned his seat at the Council Board of the Physical Society. He is therefore no longer Honorary Secretary of that Society.

THE ANALYSIS OF BEARING-METAL ALLOYS. WITH A NEW VOLUMETRIC METHOD FOR DETERMINING COPPER.*

By W. E. GARRIGUES.

(Concluded from p. 41).

New Process for Copper.

THIS depends on the precipitation of the copper as cuprous thiocyanate according to Rivot, and the volumetric determination of the combined thiocyanic acid.

The solution is evaporated with as little sulphuric acid as possible to expel any volatile acids; warm gently after dilution, and add sulphurous acid until its presence is plainly apparent by odour. Precipitate with an alkali thiocyanate. When the liquid settles perfectly clear, which is promoted by further warming, filter and wash well with water.

Transfer the filter and contents back to the beaker in which the precipitation was effected, boil with a measured excess of standard caustic alkali, a few minutes is sufficient, then cool and dilute to a convenient bulk (200 c.c.). After passing through a dry filter, take of the filtrate a quantity equal to one-half the bulk of the original solution, and titrate to neutrality with standard acid and methyl orange.

The rationale of the process is that caustic alkali converts cuprous thiocyanate into cuprous hydroxide and alkali thiocyanate, the latter body being neutral to methyl orange. The reaction proceeds quantitatively according to the equation—



with all quantities of copper, one equivalent of caustic soda equalling one of copper. No requirement of an empirical standard is one of the advantages of the process. The writer claims for it greater accuracy than the iodide method is capable of.

Antimony is determined by Mohr's method: titration with iodine in alkaline solution, antimonious acid being previously reduced by the method of Gooch and Gruener (*Am. J. Sci.*, xlii., 233). This is very much to be preferred to the oxidimetric method again recently advocated by Thompson (*J. Soc. Chem. Ind.*, xv., 255), or to any attempt at separation from tin.

Lead is determined as sulphate, filtering on a Gooch with a paper disc, drying and weighing. In this manner the determination is capable of great accuracy, the drying being much better than ignition.

Tin is weighed as oxide, or in presence of antimony is determined by difference in weight.

Arsenic by Lundin's distillation process, as described by Blair in his work on iron analysis.

Phosphorus as magnesium pyrophosphate.

General Considerations.

When an alloy is treated with nitric acid, a more or less white residue remains insoluble. This contains all the tin, part of the antimony, iron, arsenic, phosphorus, copper, and lead, the two last, of course, only in small amount, though the copper may amount to several milligrams. No method of treatment has been found which gives this residue free from iron and copper. If all excess of nitric acid be evaporated, and the residue again taken up with dilute nitric acid, it is possible to retain all the iron in the residue, provided the tin is present in fair amount. The same applies also to the arsenic and antimony, though it is probably unsafe to count on this. In no event can the insoluble residue from a brass or bronze be considered sufficiently pure to weigh as stannic oxide. We separate the iron, copper, and lead from the tin and

* Read before the Chemical Section of the Engineers Society of Western Pennsylvania. From the *Journal of the American Chemical Society*, vol. xix., No. 12, December, 1897.

antimony by fusing with sodium thiosulphate and dissolving in water, preferring this to attempting to make the usual alkali sulphide separation in solution with all the copper and lead. The thiosulphate is a neater and quicker flux than sodium carbonate and sulphur.

If to save time in determining tin alone, when phosphorus, arsenic, and antimony are absent, it is desired to obtain the weight of stannic oxide by difference, the nitric acid insoluble residue may be ignited and weighed, fused with thiosulphate, dissolved in water, and the sulphides of lead, copper, and iron remaining removed by filtration. The filter is then moistened with nitric acid, and ignited in the same crucible (the fusion does not alter its weight perceptibly), subtracting the weight so found from the total weight of the residue. The difference is taken as stannic oxide.

A slight error must, however, be noted here: any lead present was weighed first as oxide, and afterwards subtracted partly or wholly as sulphate. It may be present to an extent that will make this error serious. If, on the other hand, the sulphides are not moistened with nitric acid, the iron is very likely to burn only to the magnetic oxide, where it was at first weighed as the peroxide, while the condition of the lead is indefinite.

In the case of a complete analysis of an alloy containing zinc, it is a great help to get all the iron out of the way at this early stage, and as the copper and lead must be returned to the main solution, it will be more troublesome to separate these, after igniting the sulphide precipitate, than to precipitate the tin directly as sulphide from the solution of the fusion, and converting to oxide for weighing. The latter also has the advantage that it is applicable in the presence of phosphorus.

Hempel (*Ber. d. Chem. Ges.*, xxii., 2478), referring to the separation of phosphorus from tin, states that some of the phosphorus invariably comes down with the hydrogen sulphide precipitate of the tin. Whether or not the same applies to the sulphide precipitate, obtained as a result of acidulating the dissolved fusion, we have not ascertained, but it seems probable that it would not. At any rate the amount is too small to practically affect a tin determination in phosphor-bronze. Arsenic, however, will largely remain with the ignited tin precipitate unless previously separated.

When the nitric acid filtrate from the insoluble residue is evaporated with a little sulphuric acid to take out the lead, there results a further precipitation of antimony. The lead sulphate cannot be removed by solution in ammonium acetate, as the antimonious acid is likewise affected. A large excess of sulphuric acid prevents this second precipitation, but its presence is undesirable for the separation of copper as thiocyanate. A little tartaric acid added to the diluting water will also prevent it, and has, moreover, no effect on the copper precipitate, but this may cause the re-solution of a trace of lead. For ordinary purposes this amount is negligible, but at any rate it may be very easily recovered and added at a later stage.

Alloys containing antimony are usually poor in copper and free from zinc; exceptions, however, must be allowed for, and in the presence of both zinc and antimony the precipitation of copper as thiocyanate is not advisable, as the scheme thereby becomes complicated. Instead, the excess of sulphuric acid is used to prevent the precipitation, with the lead sulphate, of any antimony that remains in the nitric acid solution. In the filtrate from the lead sulphate the antimony and copper are thrown down together as sulphides, and separated with caustic soda. In the residue the copper may, of course, be determined by the thiocyanate process, but as it is free from any other metals it is directly available for titration by the iodide method. In the filtrate from the sulphides the zinc is obtained as phosphate. It should be remembered that if great accuracy is desired in the figure for zinc, the sulphide precipitate, after removing the antimony, is to be dissolved and again precipitated to insure against loss of zinc.

As a result of converting the mixed sulphides of tin and antimony into oxides for weighing, a slight error is sustained owing to the fact that the two sulphides require different treatment to give accurate results. By treatment with fuming nitric acid combined sulphur is oxidised completely, and the full heat of a good Bunsen burner will convert all pentoxide of antimony to tetroxide without any trouble, but while the tin precipitate requires a white heat to drive out the last traces of water, antimony will not stand this heat. If, therefore, the Bunsen flame be used, and one-tenth per cent be subtracted from each twenty per cent of tin found, the result is better than if the actual weight obtained is taken.

As the sulphides of tin and antimony are recovered from the dissolved fusion, they are mixed with considerable free sulphur, which cannot be completely oxidised with fuming nitric acid in one evaporation. For the most careful work washing the dried sulphides with carbon disulphide is necessary, but ordinarily, careful ignition will answer. After evaporating the mass to dryness with fuming nitric acid, and burning at a temperature just sufficient to ignite the sulphur, the residue is again moistened with fuming nitric acid for final ignition. This is important. The paper is, of course, to be consumed separately. A well-rounded inverted lid is used as a cover during the action of the acid.

When the ignited oxides of tin and antimony are dissolved by fusion with caustic (or carbonated) alkali, and obtained as higher chlorides in acid solution, potassium iodide reduces only the antimonious salt. We prefer to boil down twice to about fifty c.c. to insure the expulsion of the liberated iodine. We have found the colour of the liquid not a safe guide in practice, except that if perceptible deepening of the yellow takes place when approaching the bulk mentioned, the liquid must be immediately diluted, and the second evaporation not carried quite so far. Failure to do this will result in loss of antimony by volatilisation.

The separation with hydrogen sulphide of copper from zinc in hydrochloric acid is, according to Fresenius, rarely perfect. On testing a number of copper sulphide precipitates from solutions of brass containing fifteen c.c. strong hydrochloric acid in a bulk of 200 c.c. of liquid, we have never failed to find zinc in appreciable quantity. The precipitation as thiocyanate is, on the other hand, absolutely free from this source of error, and if the volatile acids are expelled with as small a quantity of sulphuric acid as possible, the amount of copper that escapes precipitation is infinitesimal. The precipitate cannot be dried and weighed with anywhere near the accuracy one might be led to suppose from the single experiment quoted by Fresenius. Such a procedure is quite out of the question.

The excess of ammonium thiocyanate exerts no deleterious action on the zinc precipitation with ammonium phosphate in the filtrate, and methyl orange is available as indicator for exactly neutralising the solution. When potassium thiocyanate is used the zinc precipitate is highly contaminated, so much so that it fuses at a low heat to a clear liquid, the results being entirely without value.

The Analysis of Bronze and Brass (Antimony being Absent).

Oxidise one grm. with strong nitric acid, and evaporate on the water-bath to complete expulsion of the acid. Take up with fifty c.c. three per cent nitric acid and filter after boiling five minutes. Ignite filter and contents together in a porcelain crucible, grind the residue with a thick glass rod, and fuse with a liberal addition of sodium thiosulphate. Leach out with a little water, digest until the liquid is clear yellow, and remove the precipitated sulphides by filtration. In the absence of arsenic, determine tin in the filtrate by acidulating with hydrochloric acid, filtering out the sulphide of tin, and washing with ammonium acetate. Ignite at low temperature, together

with the filter, raising the heat gradually after all free sulphur has been consumed, and finishing with a blast to constant weight.

The mixed sulphides of lead, copper, and iron, recovered from the fusion, are dissolved by boiling with twenty per cent nitric acid, from which solution the iron is thrown down by ammonium acetate. The filtrate is added to the main solution, and the whole evaporated with as little sulphuric acid as possible, the lead sulphate collected on a Gooch crucible and weighed.

In the filtrate the copper is thrown down as thiocyanate, and its value determined by the method described. The zinc is then obtained from the neutralised filtrate with ammonium phosphate, observing the precautions mentioned.

Phosphorus is determined in a separate portion by heating some time with aqua regia, passing hydrogen sulphide into the alkaline solution and filtering. The phosphorus is then obtained with magnesia mixture as usual. If qualitative tests have shown presence of arsenic the magnesia precipitate is dissolved in hydrochloric acid, arsenic reduced by boiling with sulphurous acid, and precipitated with hydrogen sulphide. In the filtrate the phosphorus is obtained with molybdate, and the yellow precipitate weighed, after collecting on a Gooch crucible with a paper disc.

Arsenic is determined in a separate portion by Lundin's method, just as described by Blair, except that we deem one evaporation to dryness with sulphuric acid sufficient.

In the tin determination, arsenic is separated from the solution of the thiosulphate fusion with magnesia mixture, the tin being then obtained in the filtrate as described.

In the analysis of brass, a more rapid, but not so accurate, determination of the copper and zinc may be obtained by using the iodide method for the former, and the ferrocyanide method for the latter. The foregoing process is carried out to the point of obtaining the filtrate from the lead sulphate. This is mixed with about ten c.c. hydrochloric acid, and the copper precipitated by boiling a few minutes with strips of sheet aluminium. For a neat manner of treating this copper, consult Low's paper already referred to.

The filtrate is ready for titration of zinc with ferrocyanide according to Stone.

The Analysis of White Metal (Antimony being Present).

Proceed as in analysis of bronze up to the point of evaporation with sulphuric acid to remove lead, but in this case use about fifteen c.c. of acid instead of about two. Dilute with eighty-five c.c. water, and filter without unnecessary delay or boiling. Dry and weigh the lead sulphate.

Copper and antimony are thrown down in the solution with hydrogen sulphide, and after boiling the filtrate free from the gas, and oxidising any fine sulphur with nitric acid, zinc is obtained as phosphate in the neutral solution.

The sulphides of copper and antimony are washed from the filter back into the beaker, boiled with a little caustic soda which is passed through the filter, and the filtrate now containing the antimony is added to the solution of the thiosulphate fusion of the nitric acid insoluble residue. The remaining copper sulphide is dissolved by boiling it in 1:20 sp. gr. nitric acid, when it is neutralised and the copper determined with potassium iodide and thiosulphate.

The alkaline solution, containing now all the tin and antimony, is acidulated with hydrochloric acid, and the mixed sulphides filtered out. Ammonium acetate is used for washing as before, but the paper is separated from the dried precipitate and burned alone, followed by the precipitate itself. Ignite in the manner already described for antimony and tin sulphides in the presence of free

sulphur. Having obtained the weight of the mixed oxides, it now remains only to determine the antimony, which is attained quite accurately, as follows:—

Grind the residue in the crucible, and pour the powder into a silver crucible containing about six grms. of previously fused caustic potash. Re-weigh the porcelain crucible to ascertain the amount of the residue actually taken for antimony, of course making correction for the difference later.

When fused, dissolve in water, add five grms. tartaric acid, and dilute hydrochloric acid to neutrality. Wash into flask, and add five c.c. strong sulphuric acid and one and a half grms. potassium iodide, keeping the solution down to a bulk of 100 c.c. as nearly as possible. Boil down rapidly to about fifty c.c., observing the precautions already referred to, dilute with boiling water to about 100 c.c., and boil down again to about fifty c.c. The antimony is now all reduced.

Cool, add a pinch of dry phenolphthalein and caustic soda to alkalinity. Discharge the red colour with dilute hydrochloric acid, cool again, add starch paste and fifty c.c. saturated sodium bicarbonate, and titrate with decinormal iodine solution to appearance of the blue colour. We find that by working in this manner one c.c. of the iodine is equivalent to 0.00615 gm. antimony. Theory requires 0.0060.

The tin is obtained by the difference in weight, calculating the antimony to Sn_2O_4 . From every 20 per cent tin so obtained, subtract one-tenth per cent, as explained previously.

In conclusion, we may add that any scheme which ignores the antimony passing into the nitric acid solution when the alloy is oxidised, is not worth considering. Working on one gm. it amounts to from three to four per cent of antimony, calculated on the alloy. Some results point to the feasibility of making correction for this solubility, instead of holding back the main precipitate for it.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, January 20th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

MR. OSCAR GUTTMANN was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. W. M. Bailey, 13, Green Lane Road, New Evington, Leicester; A. J. B. Cooper, Grimstone Lawn, Ealing, W.; John R. Don, D.Sc., M.A., Waitaki, Oamaru, N.Z.; F. W. Dootson, M.A., Bryn, Glisson Road, Cambridge; John Glaister, M.D., 4, Grafton Place, Grafton Square, Glasgow; David Homfray, B.Sc., 6, Dartmouth Row, Greenwich, S.E.; A. F. McEwen, 43, Gilmore Road, Lewisham, S.E.; T. H. Pope, South Street, Ponders End.

Election of Foreign Members.

A ballot for the election of Foreign Members was held, and the following were subsequently declared duly elected:—Professors S. Arrhenius, Th. Curtius, A. P. N. Franchimont, W. Körner, W. Markownikoff, N. A. Menshutkin, H. Moissan, W. Ostwald, F. M. Raoult, I. Remsen, W. Spring, L. J. Troost, P. Waage, J. D. van der Waals.

ANNOUNCEMENT BY THE COUNCIL.

The PRESIDENT said that he was authorised by the Council to make the following statement to the Society:—

The Council of The Chemical Society have received the following Memorial and enclosures.

Letter addressed to the Fellows by Messrs. Harden and Hartog.

Owens College, Manchester, October, 1897.

DEAR SIR,—We desire to ask if you will give your support to the enclosed Memorial to the Council of The Chemical Society, requesting them to propose such alterations in the Rules of the Society as will enable members who reside in the country to record their votes in the election for officers and members of the Council without personal attendance at the General Meeting.

The reasons for the change which we desire are explained in the Memorial itself, of which a draft is enclosed. That the change is practicable is shown by the fact that in Societies like the Society of Chemical Industry, the Institution of Civil Engineers, and the Institution of Electrical Engineers, each containing several thousand members, the regulations allow votes to be recorded by post, as we suggest, and the arrangements are such that the secrecy of the ballot is preserved. Should you approve of it, we should be much obliged if you would kindly sign and return the draft Memorial under cover to one of the undersigned at your early convenience.

We are, dear Sir,
Faithfully yours,

ARTHUR HARDEN.
PHILIP HARTOG.

MEMORIAL.

To the President and Council of The Chemical Society.

GENTLEMEN,—We the undersigned, Fellows of The Chemical Society, desire to draw your attention to the great change in constitution which has taken place since the Society was originally formed, and to the desirability of making some alteration in the Bye-laws, so as to correspond with this change. Whereas the Society was originally composed in the main of chemists living in London or its immediate neighbourhood, and until 1871 was called on its official publication The Chemical Society of London, since that date it has been called by the more general name (given in the Charter) of The Chemical Society, and at the present time the vast majority of the Fellows reside in other parts of the kingdom.

Under the existing regulations it is, however, practically impossible for this majority to take any share in the government of the Society, since, under Bye-law V., the vote of each member at the election of Officers and Council must be recorded *personally*, and may not be sent by post.

It is, of course, recognised that a custom has grown up of choosing a certain proportion of the Officers and Council from among the Fellows who reside in the provinces, but this practice cannot be regarded as a sufficient remedy for the defect in the existing Bye-law, and while we do not wish in any way to express dissatisfaction with the policy of the present or of past Councils, we feel strongly that the Council should be elected by the whole Society, and not by a mere fraction of the Fellows. In this feeling we venture to think that you will concur.

We trust, therefore, that you will see your way to propose to the Society at an early date an alteration of Bye-law V. in the sense we desire, and have the honour to remain, Gentlemen,

Your obedient Servants,
(Signed by 540 Fellows).

Letter received by the Secretaries, together with the Memorial and Enclosures.

Owens College, Manchester, December 16th, 1897.

GENTLEMEN,—We beg to forward herewith a Memorial addressed to the President and Council of The Chemical Society, together with the signatures of over 500 Fellows appended thereto. We also enclose a list of the signatures divided into two sections:—(a) A list of the signatures appended to the Memorial in the first instance—

i.e., before it was circulated among the Fellows of the Society residing in the United Kingdom generally; (b) a list of signatures appended subsequently. With reference to the substance of the Memorial we wish to make one statement. We are aware that in the Charter (on page 8 of the printed text) it is provided that "at all general meetings and meetings of the Council, the majority present, and having the right to vote thereat respectively, shall decide upon the matters propounded at such meetings"; hence in order to render legal the change desired, it would probably be necessary to obtain a Supplemental Charter, allowing members not actually present at meetings to vote by means of balloting papers. It is in this connection material to point out that in order to obtain a revision of Bye-laws precisely similar to that now desired by Fellows of our own Society, a supplemental Charter was quite recently obtained by the Institution of Civil Engineers. We beg to request you to lay the accompanying Memorial and enclosures, and the present letter, before the President and Council of The Chemical Society, and have the honour to remain, Gentlemen,

Your obedient Servants,

ARTHUR HARDEN.
PHILIP HARTOG.

The Secretaries of The Chemical
Society.

Owing to the action of the Bye-laws Committee, which was appointed some time ago, the Council is in a position to give an immediate answer to the Memorialists.

The question of the power of the Society to alter the mode of election of the Officers and Council having arisen, the Bye-laws Committee requested the solicitors of the Society, Messrs. Wilson, Bristows, and Carpmael, to submit the following case for legal opinion:—

Case for the Opinion of Counsel.

The opinion of Counsel is desired by the President and Council of The Chemical Society under the following circumstances:—

A memorial is being extensively signed by the Fellows of The Chemical Society, addressed to the President and Council of such Society, asking that the Bye-laws may be altered so as to allow the votes at the General Annual Meetings to be given by proxy. A printed copy of such Memorial is sent herewith.

The attention of Counsel is directed to page 8 of the printed copy of the Charter of The Chemical Society, which is sent herewith, and also to pages 16 and 17 of such document. On the latter of which pages will be found the present Bye-laws on the subject.

Counsel is desired to advise the President and Council of The Chemical Society whether, under their Charter, they have power to alter the Bye-laws so as to provide for the votes at General Meetings being given by proxy.

The following is the opinion of Counsel:—

Copy of Opinion of Mr. COZENS-HARDY, Q.C.

I am of opinion that the Charter (p. 8) prohibits voting by proxy, and that any Bye-law framed for the purpose of allowing voting by proxy would be repugnant to the Charter (p. 9) and invalid.

HERBERT H. COZENS-HARDY.

7, New Square, Lincoln's Inn,
December 6, 1897.

This opinion of Mr. Cozens-Hardy, Q.C., makes it clear that the Council of the Society have not the power to propose such an alteration of Bye-law V. as the Memorialists request.

The suggestion made by Messrs. Harden and Hartog in the letter addressed to the Secretaries along with the Memorial, that "it would probably be necessary to obtain a Supplementary Charter allowing members not actually

present at meetings to vote by means of balloting papers," opens up questions outside the terms of the Memorial, which will necessitate further legal advice. The whole subject is receiving the most careful consideration of the Council.

Of the following papers those marked * were read:—

*1. "The Preparation of Pure Iodine." By BEVAN LEAN, D.Sc., B.A., and W. H. WHATMOUGH.

Stas, in his "Nouvelles Recherches sur les Lois des Proportions Chimiques," states that he was only able to find two methods of preparing iodine free from chlorine and bromine. One consisted in precipitating by water iodine dissolved in potassium iodide, the other depended on the decompositions of iodide of nitrogen by heat. Perhaps the main difficulty attached to these methods was the desiccation of the iodine and the removal of hydriodic acid. Neither in his published memoirs nor in his laboratory note-books has Stas stated how he assured himself that his "iodine" was free from other halogens.

A few months ago the authors observed incidentally that no iodine is set free when cuprous iodide is heated, even at its fusion-point, in a current of carbonic anhydride, although it is readily evolved when cuprous iodide is heated in air, oxygen, nitric oxide, or nitrogen peroxide. The action is represented by the equation $\text{Cu}_2\text{I}_2 + \text{O}_2 = 2\text{CuO} + \text{I}_2$. No iodine is evolved when cuprous iodide is heated in a vacuum.

The authors have examined the usual methods of preparing cuprous iodide. When a solution of cupric sulphate and a soluble chloride, bromide, or iodide is saturated with a sulphurous acid, cuprous iodide, bromide, and chloride may all be precipitated; but there is so great a difference in their degree of solubility that, by securing a proper dilution, it is probable that cuprous iodide can be prepared free from cuprous bromide or chloride. A mixture of cupric sulphate and ferrous sulphate is still less liable to precipitate cuprous bromide and chloride along with cuprous iodide than cupric sulphate saturated with sulphurous acid. The authors have found that cuprous iodide can also be prepared by sprinkling iodoform in small quantities at a time upon a hot surface of copper. On account of the difference in the properties of chloroform, bromoform, and iodoform, it is probable that, by this method also, cuprous iodide can be prepared entirely free from bromide or chloride. If, moreover, the cuprous iodide is fused in a current of carbonic anhydride, or *in vacuo*, it can be freed completely from moisture.

Iodine is most conveniently prepared from cuprous iodide by heating it in a stream of dry air at 220° to 240° and condensing the vapours upon a cold surface. Although the greater portion of the iodine in a given quantity of cuprous iodide is quickly expelled, it is not easy to expel the whole; after heating 17101 grms. at 400° for eighteen hours, 0.15 per cent of the iodine was still undecomposed—otherwise the relation $\text{Cu}_2\text{I}_2 : 2\text{CuO}$ could have been utilised for the exact measurement of the atomic weights of copper, iodine, and oxygen. The authors are making further experiments upon this point. The action of air upon cuprous iodide is not dependent upon the presence of moisture. This is established by sealing cuprous iodide in glass tubes in the presence of phosphoric anhydride.

Iodine liberated as described from cuprous iodide at 240° leaves absolutely no residue when volatilised at 75° . If examined spectroscopically, no evidence of the presence of copper can be found. The melting-point (uncorrected) is 112.5° to 114° .

Whether such iodine is as pure as that prepared by Stas or not, it appears desirable to re-determine the atomic weight of the element prepared by this method.

DISCUSSION.

Dr. SCOTT remarked that Stas depended on distillation two or three times with anhydrous baryta, and not on calcium nitrate, for the complete removal of water from his iodine. This reagent had also the great advantage

of removing any traces of hydriodic acid, the calcium nitrate being only used for the removal of the greater part of the water. Although Stas does not give his methods of testing his iodine for traces of chlorine and bromine, his concluding remarks in that section of his paper evidently show that he had no doubts as to its purity. The marvellous agreement of his results with those of Marignac, who found 100 grms. of silver gave 217.5334 of silver iodide, whilst Stas found in his complete synthesis 217.5335, will probably convince most chemists that both were dealing with the same chemical substance.

Mr. R. J. FRISWELL asked if the author had observed the crystalline form and appearance of the sublimed iodine. He had himself a very interesting experience in this matter with iodine recovered from methyl iodide by Nicholson's method of recovering the iodine, used in methylating rosaniline by mixing the sulphuric acid and sodium bichromate with the waste iodine liquor, the precipitated iodine being washed, pressed, and fused under sulphuric acid.

For many years he (Mr. Friswell) had noticed that at times the sulphuric acid used for the fusion contained a mass of minute, violet-brown, glistening plates, but every attempt to collect them failed, the least dilution of the acid causing them instantly to disappear. After some time, however, nearly a pound of a paste or magma of these crystals was obtained in the following way:—The acid was allowed to dilute itself by the absorption of water from the air and then run off from the crystals; the magma of crystals was thrown on to a vacuum filter and washed with sulphuric acid of continually increasing dilution, and at length with water; the paste was then squeezed, and, while moist, sublimed in a beaker covered with a loose glass plate at a temperature of 35 to 40° .

At first, the ordinary violet plates of iodine appeared, with a little water, but these soon vanished, and intensely black, adamantinite, solid rhombic (?) crystals appeared. A brownish earthy residue was left. The crystals were of considerable size, a cubic m.m. or so in content. He thought that since these had been obtained from iodine continually in use for many years, during which time some tons of iodine had passed through the operation, they might be the companion element which has been suspected. Prof. Ramsay was asked to examine the crystals and the paste from which they had been sublimed, but found that the adamantinite crystals gave no residue on sublimation and showed 99.84 per cent iodine, the deficit being probably due to a slight accidental loss. On sublimation in a vacuum, both the plate-like crystals which first sublimed and iodine purified in the usual way left slight yellowish residues, but these adamantinite crystals left none. They were, therefore, pure iodine. He wished to know whether the author's substance resembled these crystals in any way.

The PRESIDENT mentioned, as indicating the general trustworthiness of Stas's observations, that he had recently confirmed Stas's remarkable statement, that, when pure, solid iodine is opaque to light. He inquired whether Dr. Lean had made any experiments with palladium iodide.

Dr. LEAN, in reply, said that he had not noticed anything anomalous in the crystallisation of the iodide. He had observed, however, that his iodine had a black and not a deep violet colour, and that it emitted no visible vapour at the ordinary temperature. Stas had made a similar observation.

The reason he had not made most of his experiments with palladium iodide was that it was so much more expensive than cuprous iodide. He had, however, made a few experiments which showed that iodine was very readily liberated when palladium iodide was heated in air.

*2. "Derivatives of Bromotolylhydrazine." By J. T. HEWITT, M.A., D.Sc., and F. G. POPP.

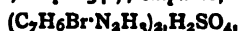
This is a continuation of the work of one of the authors on ortho-substituted phenylhydrazines. The hydrazine was prepared from the amido-compound by the

method of Victor Meyer and Lecoq (*Ber.*, 1883, xvi., 2976), an alteration being made by pouring the solution of the diazonium salt into the stannous chloride solution. The free hydrazine and the following salts and derivatives are described:—

Bromtolylhydrazine, $C_6H_5BrMe(N_2H_3)$, 1:3:6, colourless needles, m. p. 91° ; *hydrochloride*, $C_7H_6Br \cdot N_2H_3 \cdot HCl$, colourless needles, m. p. 190° ; *nitrate*,—



colourless plates, m. p. 154° ; *sulphate*,—



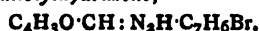
colourless needles, m. p. 201° ; *oxalate*,—



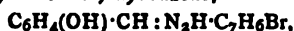
small colourless prisms, m. p. about 150° . *Acetylbromtolylhydrazine*, $C_7H_6Br \cdot NH \cdot NH \cdot CO \cdot Me$, colourless prisms, m. p. 124° . *Bromtolylsemicarbazide*,—



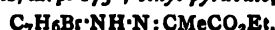
small colourless crystals, m. p. 163° . *Bromtolylallylthiosemicarbazide*, $C_7H_6Br \cdot NH \cdot NH \cdot CS \cdot NH \cdot C_3H_5$, colourless prisms, m. p. 136.5° . *Bromtolylphenylthiosemicarbazide*, $C_7H_6Br \cdot NH \cdot NH \cdot CS \cdot NH \cdot C_6H_5$, small prisms often grouped in radiating clusters, m. p. 142° ; the freshly prepared substance usually has a somewhat lower melting-point, and this may possibly be a case of isomerism similar to that observed by Marckwald with diphenylthiosemicarbazide (*Ber.*, 1892, xxv., 3098). *Furfuraldehydebromtolylhydrazones*,—



needles, m. p. 87° . *Benzaldehydebromtolylhydrazones*, $C_6H_5 \cdot CH : N_2H \cdot C_7H_6Br$, rhomboidal plates, m. p. 84° . *Salicylaldehydebromtolylhydrazones*,—



needles, m. p. 109° , and the following bromtolyl hydrazones of:—*pyruvic acid*, $C_7H_6Br \cdot NH \cdot N : CMeCO_2H$, yellow crystals, m. p. 175° ; *ethyl pyruvate*,—



tufts of needles, m. p. $84-85^\circ$; *potassium pyruvate* (with 3 mols. of water), $C_7H_6Br \cdot NH \cdot N : CMeCO_2K \cdot 3H_2O$; *ammonium pyruvate*, $C_7H_6Br \cdot NH \cdot N : CMeCO_2NH_4$ (both these salts are soluble in hot water, sparingly, however, in cold water); *lead pyruvate*,—



a pale yellow precipitate.

The effect of heat on the potassium and lead salts is to furnish potassium or lead bromide as well as basic and acidic or phenolic decomposition products. The reactions are being further examined.

*3. "Researches on the Terpenes. I. On the Oxidation of Fenchene." By JOHN ADDYMAN GARDNER and GEORGE BERTRAM COCKBURN.

The authors give an account of their method of producing fenchene from fenchone, the oxidation products of fenchene, and pinene hydrochloride.

Fenchone, b. p. $191-192^\circ$, m. p. 6° , specific rotatory power $[\alpha] = +61.58^\circ$ was reduced to fenchyl alcohol by a modification of Wallach's method, using amyl alcohol and sodium instead of ethyl alcohol and sodium. It was obtained in hard, white crystals, m. p. 45° , specific rotatory power $[\alpha] = -13.38^\circ$.

The fenchyl alcohol was converted into fenchyl chloride by the action of phosphorus pentachloride, and this into fenchene by saponification with aniline. The bulk of the fenchene thus obtained distilled for the most part between 150° and 156° , several degrees lower than that described by Wallach, which boiled between 158° and 160° . A fraction, $152-154^\circ$, was shown by combustion to have the formula $C_{10}H_{16}$. Its specific gravity was 0.8667 at 18° , and specific rotatory power $[\alpha] = -6.46^\circ$ (not in solution). The specific gravity of Wallach's hydrocarbon was 0.864 at 20° , and it was optically inactive.

As a by-product in the action of fenchyl chloride on aniline, the authors obtained a black oil, not volatile in steam. This substance did not solidify on standing, but on distillation under reduced pressure came over for the most part at $171-173^\circ$ at 13 m.m. pressure. It was a pale yellow, thick, viscous oil, which turned black on exposure to air. Analysis showed it to have the composition $C_{10}H_{17}NHC_6H_5$. On treatment with acetyl chloride it yielded a thick, viscous oil, of the composition—



Fenchene was oxidised by heating it on the water-bath with nitric acid (1 nitric acid : 1 water). When the oxidation was complete the liquid was distilled in steam, when some acetic acid was obtained. The residue was then evaporated to small bulk, and on standing crystals of *cis*-camphopyric acid separated out. After the removal of these crystals, the oily mother-liquors were eventually distilled *in vacuo*. An acid oil and a neutral solid distilled over; these were separated by means of sodium carbonate, and the solid crystallised from alcohol. It proved to be camphopyric anhydride, m. p. 178° . The yield of camphopyric acid obtained in these experiments was about 20 per cent.

Turpentine hydrochloride was heated on the water-bath with nitric acid (2 nitric : 1 water). The oxidation was exceedingly slow, but after about ten days the bulk of the hydrochloride had disappeared. The greater part of the acid was then distilled, and contained a considerable proportion of acetic acid. The residue was then evaporated to complete dryness, dissolved in ether, and the ethereal solution extracted with sodium carbonate.

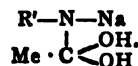
The acids extracted by the alkali were converted into insoluble lead salts. The lead salts were decomposed by sulphuric acid, and extracted with ether. On evaporating the ether an oil was obtained which, on standing, deposited crystals of camphoric acid; the purified acid melted at $202-203^\circ$, and its anhydride at 217° .

The oil left after separation of these crystals was distilled in a vacuum; an oil distilled over between $120-130^\circ$, a smaller portion at $130-150^\circ$, and a considerable amount at $180-200^\circ$, which solidified on standing. This latter portion was purified by sublimation and crystallisation from alcohol, and proved to be camphopyric anhydride, m. p. 178° .

The yield of camphoric acid from 300 grms. of turpentine hydrochloride was 18 grms., and of camphopyric anhydride 8-9 grms. The authors were unable to isolate any camphoic acid.

4. "The Action of Alkalis on Amides." By JULIUS B. COHEN, Ph.D., and CHARLES E. BRITAIN, B.Sc.

The authors have succeeded in preparing a series of compounds of the general formula, $R'NH \cdot C_2H_5O \cdot NaOH$ and $R'NHC_2H_5O \cdot KOH$, by the action of the caustic alkalis on amides under certain conditions. These substances are analogous in their mode of formation, and in properties to the alcoholates previously described (Cohen and Archdeacon, *Trans.*, 1896, lxi., 9), and probably possess a similar constitution, which may be expressed by the formula—



5. "The Formation of Monomethylaniline from Dimethylaniline." By JULIUS B. COHEN, Ph.D., and HARRY T. CALVERT, B.Sc.

When phenylnitrocarbinol (*Trans.*, 1897, lxxi., 1050) acts on dimethylaniline a violent reaction occurs, and the dimethylaniline is converted into nitrosomethylaniline, and at the same time nitrogen is evolved, and benzylalcohol and benzaldehyde are formed. As phenylnitrocarbinol readily evolves nitrous fumes on standing, and reacts in some cases like nitrous acid, this result suggested the possibility of a similar reaction occurring when nitrogen trioxide acts upon dimethylaniline. The latter action

is, however, entirely distinct from that of phenylnitro-carbinol or nitrous acid, and will form the subject of a future communication.

6. "Note on the Aluminium-Mercury Couple." By JULIUS COHEN, Ph.D., and HARRY T. CALVERT, B.Sc.

The authors find that when aluminium is amalgamated with mercuric chloride, a small quantity of chlorine is retained by the aluminium, and is probably present as oxy-chloride. The presence of the chlorine first became evident in the process of reducing a nitro-compound in neutral solution, when, instead of the free base, the hydrochloride was formed.

7. "Action of Chloroform and Alkaline Hydroxides on the Nitrobenzoic Acids." By WALTER J. ELLIOTT, M.A.

The author finds that the meta- and para-acids are reduced to the corresponding azoxy-acids, while the ortho-acid is not appreciably affected.

PHYSICAL SOCIETY.

Ordinary Meeting, January 21st, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

Prof. FITZGERALD exhibited some photographs by Mr. Preston, in illustration of the Zeeman effect, for various cases, including those of iron, cadmium, zinc, and sodium. These photographs and the method of obtaining them have already been described. The cause of doubling is now attributed by Prof. Fitzgerald to absorption by the surrounding vapour. In a particular case he examined a double line that exists in one of the photographs. Under the polariser the two lines are at first distinctly seen; but when the polariser is turned a thin line appears in the middle, and this central line is therefore circularly polarised in a direction opposite to that of the outer pair of lines. The reason for the appearance of doubling in the first position of the polariser is, that the central line is there completely absorbed out by the surrounding vapour.

Prof. OLIVER LODGE then gave a communication concerning his work on "*Electric Signalling without Connecting Wires.*"

From the nature of the oscillatory disturbances emanating from any of the customary forms of Hertz vibrator, syntony has hitherto been only very partially available as a means for discriminating between receivers. There is, in fact, so rapid a decrease in the amplitude of the vibrations that almost any receiver can respond to some extent. Discrimination by syntony is possible with magnetic systems of space telegraphy, where the magnetic energy much exceeds the electric, *i.e.*, as between two separated inductive coils; and by the use of such coils, appropriately applied, the author has been able to attain fair syntony even with true Hertz waves, *i.e.*, he has constructed spark-gap oscillators, with sufficient persistence of vibration, and syntonised resonators. The "coherer" principle can be applied to either a purely magnetic or to the Hertzian system. It was first used by Prof. Lodge in devising lightning-guards, and afterwards in his magnetic system of telegraphy by inductive circuits, each in series with a Leyden jar; a pair of knobs in near contact, or other over-flow gap, being provided in the receiving apparatus. This was the first meaning of a "coherer" in the electrical sense as used by Prof. Lodge: it referred to a single contact between two metal knobs. The term has since been extended by others to the filings-tube of M. Branly, and some confusion has arisen, for M. Branly does not consider that simple coherence and break explains fully the behaviour of his instrument.

Prof. Lodge is disposed to agree, for he finds that the resistance of almost any form of coherer varies in rough proportion to the received impulses, and that there are other peculiarities (to be mentioned later); he is therefore inclined to think that the action cannot after all be entirely explained as due to mere "welding," but that there is something more to be learnt about it. The sensitiveness of a coherer depends upon the number of loose contacts; it is a maximum for a single contact, *i.e.*, for a needle-point lightly touching a steel spring. With this sensitive coherer, hardly any "tapping-back" is required for decoherence, but it wants delicate treatment when properly adjusted, and the greatest current through it should not approach a milliampère. On the other hand, a Branly tube rather improves under rough treatment; in such a tube the author prefers to use iron filings in the best possible vacuum: brass, too, is very good, but rather less easy to manage. Aluminium is thoroughly bad, and gold, for an opposite reason, will not work—its surface is too clean. Points, or small surfaces for making contact with the filings, are better than large surfaces. The usual method of connecting the coherer across the gap of an ordinary Hertz receiver, in parallel with the telegraph instrument and battery, has the unavoidable objection that they shunt away part of the received oscillations. With the sytonic receiver of Prof. Lodge, which contains no gap, but a closed wire coil instead, the difficulty no longer exists; for the coherer can now be in series with the detecting instrument, and, in so far as these obstruct the oscillations, they may be shunted out in various ways, as the author describes. The main feature of his new syntonised vibrators is this self-inductance coil, whose function it is to prolong the duration of the oscillations, and thereby to render syntony possible. Although such a coil acts disadvantageously in so far as it possesses resistance, the resistance does not increase so fast as the self-induction. The coil should consist of thick copper of highest conductivity, and it should have maximum inductance for given resistance. For similar reasons, the capacity areas should also be of highest conductivity, their dimensions should increase outwards from the spark-gap, as triangles. The receiver must have no gap, it should be accurately bridged over when a transmitter is used as receiver. The limit of speed of response depends upon the telegraphic instrument. Dr. Muirhead adapted a syphon-recorder to the purpose, because it is one of the quickest responders; he arranged it so that it could be used with intermittent currents, direct. Under these intermittent impulses the syphon trembles, and instead of the ordinary syphon-signals, the slip is marked with dots and dashes. Constant mechanical tremor is usually employed for decoherence, but the author finds that decoherence can be brought about by electrical means, without any mechanical tremor, by connecting the coherer momentarily to a circuit less effective as a collector than that of the proper capacity areas of the syntonised receiver. The battery and galvanometer detector-circuit may be used for this purpose, the coherer being momentarily connected to it, and while so connected letting it experience an impulse from a distance. Prof. Lodge has designed a revolving commutator, by means of which the coherer can be rapidly changed over from the resonating circuit to the instrument circuit, and finally to the "tapping-back" apparatus. A coherer is more sensitive when thus isolated and exposed to the full influence of the received oscillations; the subsequent detection of the effect by altered connections is very convenient for laboratory measurements. A diagram of a series of plotted measurements showed that the resistance of an undisturbed filings-tube is approximately a direct function of the intensity of the received stimulus, whether successive stimuli increased or decreased in strength. This electrical process of "tapping back" is to be depended upon, but the process long continued fatigues the tube until a mechanical shake is employed to restore it. Large size apparatus, made by Dr. Muirhead for actual

distant syntonic work, was exhibited, and means were shown for protecting and isolating the coherer when its receiving areas were being used as emitters; also a switch used for changing at one movement all the connections from "sending" to "receiving."

Prof. THRELFALL said he had come to the same conclusion as Prof. Lodge as to the advisability of diminishing the number of contact points in the coherer. He had endeavoured to produce longer and more persistent waves, and thus to set afield greater effective energy. It was desirable to keep the waves as parallel as possible. He thought there was some probability that the wave-fronts could be altered and rendered more comfortable by a process of diffraction.

Mr. RUTHERFORD also had found it best to work with long waves. He fully appreciated the advantage of increasing the capacity of the oscillator by extending the surface of the metallic plates.

Mr. CAMPBELL-SWINTON asked whether experiments had been made to verify Hertz's results as to the influence of reflectors behind oscillators and receivers. He had found them disadvantageous. A single wire behind either apparatus seemed partially to annul the effect. He also asked whether Prof. Lodge had observed the extraordinary sensitiveness of coherers to small changes of current in neighbouring circuits.

Prof. LODGE, in reply, said he had observed the sensitiveness to slight sudden variations of current referred to by Mr. Campbell-Swinton; for instance, when electric lamps were switched on and off. The effect of mirrors had been studied by Prof. Fitzgerald. They required to be of large dimensions as compared to the oscillator and receiver, otherwise the true reflections were not obtained.

Dr. SILVANUS THOMPSON afterwards exhibited a Tesla Oscillator. This apparatus is intended to replace the two induction coils and spark-gap arrangements used by Mr. Tesla for high frequency experiments. It consists of an induction coil, with a separate self-inductance coil in the primary circuit. This self-inductance coil is also used as an electromagnet for the separate interrupter of the primary circuit. A condenser is connected between one end of the primary coil and one terminal of the interrupter, so as to include both of them between its terminals. The primary is a single turn of copper strip six inches wide. The secondary is one layer of thick wire, each turn separated from the next by an air space. The supply current, about half an ampere, may be taken from the electric light mains at almost any voltage from 50 to 200, direct or alternating.

Prof. LODGE said it would work quite well at 10 volts. He pointed out also that if the straight discharge rods at the spark-gap were free to slide, the discharge drove them back into their sockets.

Prof. FITZGERALD said it was stated at Toronto that the spark was broken at the interrupter when the condenser was charged, and that by the time the condenser was ready to discharge, the contact at the interrupter had been made again. It seemed to him that the condenser discharges and surges must take place at a rate far higher than the period of the mechanical movement of the interrupter. The condenser charges and discharges were very rapid. It was not what is ordinarily called the "time constant" that was involved, for that only referred to constant voltage. Here the voltage was changing very rapidly indeed.

Prof. HERSHEL asked if such an apparatus was suitable for work with Röntgen rays.

Dr. THOMPSON, in reply, congratulated Mr. Tesla upon the perfect working and compactness of his invention. The present form was not suited for Röntgen ray experiments, but Mr. Tesla had designed a special coil that was excellent for that purpose.

The PRESIDENT proposed votes of thanks, and the meeting was adjourned until February 11th.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 1, January 3, 1898.

M. van Tieghem was elected Vice-President for the year 1898.

MM. Darboux and Bornet were elected members of the Central Administrative Committee for 1898.

The retiring President, M. Ad. Chatin, announced the condition of the publications of the Académie, and the changes which have taken place among the members during the year 1897.

On the Isocyanic Ethers, and the Heat of Formation of Liquid Cyanic Acid.—Paul Lemoult.—Isocyanates obtained by the action of methyl and ethyl sulphuric ethers on cyanate of potassium give but a small return. A better way is to heat cyanate of potassium with ethylsulphate of soda; the liquid produced contains 15 per cent of pure isocyanate of ethyl. *Isocyanate of methyl* is a colourless liquid boiling at 40° and polymerising with great facility. The results of some calorimetric experiments were—Heat of combustion of 1 grm., 4718.1 cal., 4732.3 cal., 4705.4 cal.; average, 4718.6 cal. From this is deduced:—

	Heat of molecular combustion.	Heat of molecular formation.
At constant volume ..	268.9 cal.	
At constant pressure ..	269.3 "	+22.8 cal.

Isocyanate of ethyl boils at 60°, and is much more stable than the above; calorimetric experiments give—

	Heat of molecular combustion.	Heat of molecular formation.
At constant volume ..	424.2 cal.	
At constant pressure ..	424.4 "	+31 cal.

From the above results the author deduces that the heat of formation of liquid cyanic acid is +20.8 cal.

A New Cyclic Ketone, Methylcyclohexenone.—A. Behal.—Methylcyclohexenone reacts on perchloride of phosphorus with considerable disengagement of heat, and forms an intensely blue-green liquid, soluble in alcohol; it becomes brown on the addition of water and alkalis; its oxidation by means of permanganate is very decided—nothing but acetic and levulic acids are formed.

No. 2, January 10, 1898.

This number contains M. Chatin's farewell address, and a long list of prizes proposed and to be awarded, but there is nothing suitable for useful abstraction.

MISCELLANEOUS.

A Simple and Efficient Boiling-point Apparatus for Use with Low- and with High-boiling Solvents.—Harry C. Jones.—The author discusses the various forms of apparatus in use, and suggests a modification of Hite's apparatus, in which the condensed solvent is not returned directly into the boiling solution. The apparatus consists of a tube about one-fourth filled with glass beads, on which rests a platinum cylinder. The thermometer is immersed in the solvent inside the cylinder, which serves to separate the condensed solvent and boiling solution. The results obtained seem to be very satisfactory.—*Journal of the American Chemical Society*, xix., No. 12.

MEETINGS FOR THE WEEK.

- MONDAY, 7th.—Society of Arts, 8. (Cantor Lectures). "Decorative Bookbinding," by Cyril Davenport.
 — Royal Institution, 5. General Monthly Meeting.
 — Society of Chemical Industry, 8. "The Curing of Malt in Relation to Colour and Value," by J. W. Lovibond. "Clerget's Method of Estimating Cane-sugar," by A. R. Ling. "A New Modification of Clerget's Method of Estimating Cane-sugar, specially applicable to Molasses and After Products," by A. R. Ling and J. T. Baker. "Note on the Estimation of Water in Invert Sugars," by Dr. L. T. Thorne and E. H. Jeffers.
- TUESDAY, 8th.—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
- WEDNESDAY, 9th.—Society of Arts, 8. "Compensation to Workmen," by A. D. Provand, M.P.
- THURSDAY, 10th.—Royal Institution, 3. "Some Italian Pictures at the National Gallery," by Jean Paul Richter.
- FRIDAY, 11th.—Physical, 5. (Annual General Meeting). President's Address. "On Electromagnetic Induction in Plane, Cylindrical, and Spherical Current Sheets, and its Representation by Moving Trails of Images," by G. H. Bryan, M.A., F.R.S.
 — Royal Institution, 9. "The Metals Used by the Great Nations of Antiquity," by J. H. Gladstone, Ph.D., D.Sc., F.R.S.
- SATURDAY, 12th.—Royal Institution, 3. "The Structure of Instrumental Music" (with Musical Illustrations), by William H.adow, M.A., B.Mus.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Dyers' Journals.—Would any correspondent kindly oblige me with the addresses of the publishers of the leading "dyers' journals" of Germany and Italy.—J. L.

TO CORRESPONDENTS.

J. E. Morce.—The only available information is to be obtained in Prof. Dewar's papers read before the Royal Society, and his lectures at the Royal Institution.

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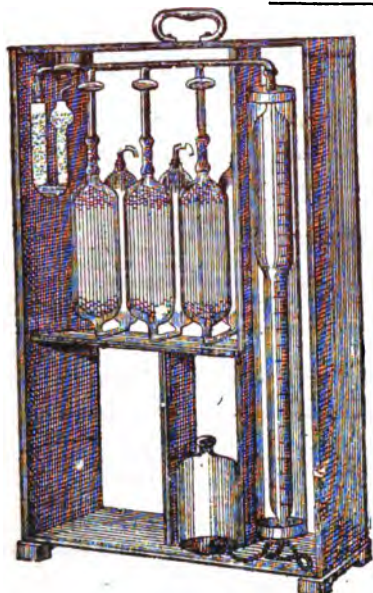
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CONTENTS.

ARTICLES:—	PAGES
The Homogeneity of Helium, by William Ramsay, Ph.D., LL.D., Sc.D., F.R.S., and Morris W. Travers, B.Sc.	61
Fergusonite, an Endothermic Mineral, by William Ramsay, Ph.D., LL.D., Sc.D., F.R.S., and Morris W. Travers, B.Sc.	64
On the Preparation and Properties of Percarbonate of Potassium, by A. von Hansen	65
On the Separation and Estimation of Iodine, Bromine, and Chlorine, by Ad. Carnot	67
The Revival of Alchemy in France, by H. Carrington Bolton, Ph.D.	69
NOTICES OF BOOKS.—The Early History of Chlorine (Alembic Club Reprints)—Researches on Molecular Asymmetry (Alembic Club Reprints)	70
CORRESPONDENCE.—Campbor and its Derivatives	70
CHEMICAL NOTICES FROM FOREIGN SOURCES	71
MISCELLANEOUS	72
MEETINGS FOR THE WEEK	72
NOTES AND QUERIES	72

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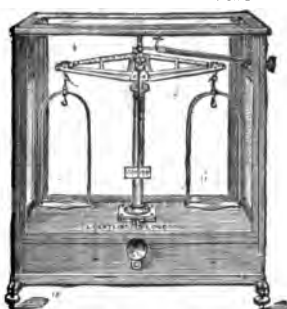
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THE HOMOGENEITY OF HELIUM.*

By WILLIAM RAMSAY, Ph.D., LL.D., Sc.D., F.R.S., and
MORRIS W. TRAVERS, B.Sc.

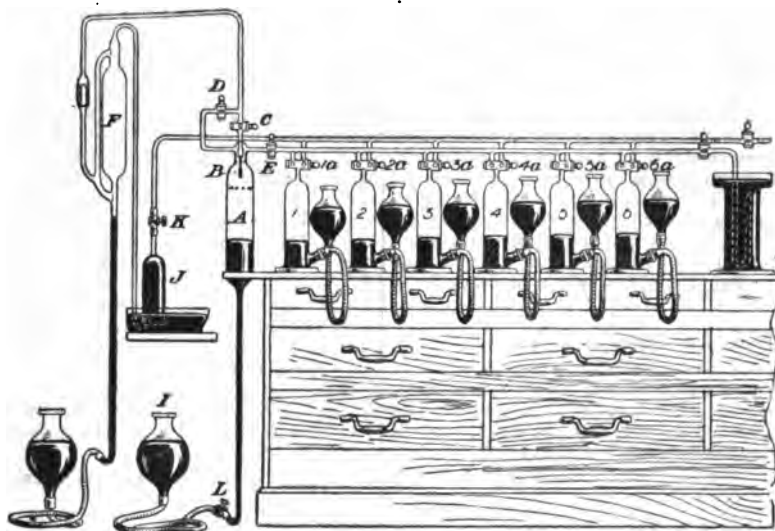
ABOUT a year ago, a paper by Dr. Norman Collie and one of the authors (W. R.) was published, bearing the title "The Homogeneity of Helium and of Argon." In that paper (*Roy. Soc. Proc.*, lx., p. 206) various reasons were adduced to show why an attempt to determine whether or no argon and helium are homogeneous was worth making. The results of the experiments at that time indicated that, while it did not appear possible to separate argon into two portions of different densities, the case was different with helium. Samples were obtained after repeated diffusion which possessed respectively diffusion

molecular magnitude. The other and more ordinary explanation of the splitting of helium into fractions of different density is that helium must be regarded as a mixture of two gases, one lighter than the other.

Since the publication of the paper mentioned, Dr. A. Hagenbach has confirmed the possibility of separating helium into portions of two densities by diffusion; and the differences in density were practically the same as those observed in the laboratory of University College (*Wied. Ann.*, lx., p. 124).

These experiments were made with somewhat over 200 c.c. of gas; but it was decided to make experiments of a similar kind on a much larger quantity of helium.

An apparatus was therefore constructed, similar in principle to the one previously employed, but on a much larger scale. The main features are shown in the illustration on p. 208 of the paper previously alluded to; but on account of the large amount of gas diffused, it was not practicable to collect it in tubes. Instead, therefore, of the bent tube *z n* of the former apparatus, the tube connected with the stopcock *z* was continued horizontally, and by means of six vertical branches it communicated with six gas reservoirs, each furnished with a two-way stopcock. It was possible with this means to cause gas



rates corresponding to the densities 2.133 and 1.874. It was there pointed out that these densities are not correct (although their ratio is probably not wrong), owing to the curious fact that the rate of diffusion of helium is too rapid for its density, *i.e.*, it does not follow Graham's law of the inverse square root of the densities. These samples of gas also differed in refractivity, and the difference was approximately proportional to the difference in density.

Towards the end of the paper, the conjecture was hazarded that it was not beyond the bounds of possibility that the systematic diffusion of what we are accustomed to regard as a homogeneous gas—for example, nitrogen—might conceivably sift light molecules from heavy molecules. It is true that the fineness of the lines of the spectrum would offer an argument in favour of the uniformity of molecular weight; but still it is never advisable to assume any physical theory without submitting it to rigorous proof. And it was thought possible that the fractional diffusion to which helium had been subjected might have had the result of effecting such a separation,—a separation, not of chemical species, but of

from any one of the reservoirs to enter the diffusion apparatus A. In order to be able to collect the gas in any desired reservoir, the delivery tube of the Töpler pump, *F*, delivered gas into a jar somewhat similar to that shown at *J*, but provided with a vertical branch, which was bent horizontally some distance up, and lay parallel to the previously mentioned horizontal tube. It, too, had six vertical branches, each of which communicated with the other limb of the two-way stopcock of each reservoir. By raising the reservoir of the Töpler pump and expelling gas into the collecting tube *J*, the gas could be transferred to any one of the reservoirs. The accompanying diagram makes it clear how the apparatus was set up.

The actual method of conducting a diffusion was as follows:—

Reservoir 1 was raised until the mercury in the diffusion jar A stood at the level of the dotted line. The clip *L* was then closed, and the stopcocks *c* and *d* opened. The Töpler pump was then worked until all gas was removed from A; the gas, if air (as at the commencement of the whole series of operations), being allowed to escape by moving the collecting jar *J*, so that it no longer covered the end of the exit tube of the Töpler pump. Stopcocks *c* and *d* were next closed, and stopcocks *z* and *6a* opened,

* A Paper read before the Royal Society, January 20th, 1898.

so that the gas from 5 entered the diffusion vessel A. By raising the reservoir belonging to 6, all gas was expelled through *x* into A, clip *L* being opened meanwhile. Reservoir 6 was now full of mercury, and all gas was in A. Stopcock *c* was then opened, and the gas in A diffused through the pipe-stem *b* (closed at one end by means of an oxyhydrogen blowpipe) into the pump. This diffusion proceeded until half the gas in A had passed into the pump reservoir *F*. Stopcock *c* was then closed, and the Töpler was worked, the diffused gas being delivered into *J*. Stopcock *6a* was then opened, and the reservoir of 6 lowered, so that the gas in *J* passed into 6. This stopcock was then shut. The contents of 5 were then transferred in a similar manner into A, and one-third of the gas was diffused into the pump. It was collected as before in 6. The diffusion jar A now contained as much gas as had been present in 5. The contents of 4 were next added; half of this was removed by diffusion and transferred to 5. The contents of 3 were added; half was diffused and transferred to 4. The contents of 2 were added; half was diffused and collected in 3. And, lastly, the contents of 1 were added, and the halt diffused collected in 2. Stopcock *d* was then opened, and the mercury in the diffusion jar A allowed to run up to the dotted line; the clip *L* was closed. All gas was pumped out of A and collected in 1; this constituted one complete round.

As it was not possible to empty the tube issuing from *J* completely of gas by lowering the reservoir of 1, and as, if not emptied, the heavy gas would have contaminated the light gas from 6 during the next round, the following method was made use of:—The gas from 6 was transferred to the empty reservoir A; and then, by lowering the reservoir of 6, mercury rose in the tube issuing from *J*, and expelled all the heavy gas in the connecting tubes into 6. The clip *x* was then closed, and by opening the stopcocks *1a* and *6a*, so that communication took place between jars 1 and 6, the small quantity of gas in 6 was transferred to 1. The apparatus was now ready for a second round.

The Fractional Diffusion of Air.

In order to test the working of the apparatus, a set of diffusions was carried out with air. After four rounds, comprising twenty-four diffusions, the light portion contained 17.37 per cent of oxygen and the heavy portion 22.03. A fairly rapid separation was thus being effected, considering the closeness of the densities of nitrogen and oxygen.

The Fractional Diffusion of Nitrogen.

A similar set of experiments was carried out with nitrogen, prepared by the action of solutions of ammonium chloride on sodium nitrite, in presence of copper sulphate. The gas was dried and passed over red-hot iron prepared by reduction of ferric oxide in order to remove any oxygen or to decompose any oxides of nitrogen which might be present. After thirty rounds, involving 180 operations, the "light" portion of the nitrogen, after purification by circulation over copper oxide, had not altered in density. It must therefore be concluded that nitrogen is homogeneous as regards the relative density of its individual molecules.

The Fractional Diffusion of Helium.

The first sample of helium employed was prepared from samarskite and cleveite. After seventeen rounds, involving 102 operations, the diffusion rates of the lighter and heavier portions were measured. The first gave a density, calculated from this rate, of 1.807, and the second of 2.128. The same gas was re-diffused until in all thirty rounds had been carried through, involving 180 operations. The light fraction now showed the density (measured diffusion rate against hydrogen) 1.816, and the heavy fraction 2.124. These gases were then circulated; the diffusion rate of the lighter portion pointed

to a density of 1.811; the heavier gas was diffused into three portions, of which the more rapidly diffusing had a "diffusion density" of 1.906, and the less rapidly diffusing of 2.032. The lightest gas of all (diffusion density = 1.811) was weighed, and had a "real" density ($O=16$) of 1.021; the mixture of the heavy products gave the real density, 2.153. The refractivity of the heavy portion, measured against helium from cleveite, undiffused, yet purified from all removable gases, which had the density (weighed) 2.076, was 1.078, the refractivity of the undiffused gas being taken as unity.

A fresh quantity of helium was next prepared from cleveite, and the former diffused samples were stored in tube-reservoirs for future use. The new helium was washed with caustic soda, but not otherwise purified. This gas was now put through fifteen rounds, comprising 90 operations, and the light portion in jar 6 was purified by circulation over magnesium and copper oxide. Its refractivity was 0.9752 of that of the uncirculated helium. Its density by weighing was 1.979. Owing to the cracking of the glass apparatus the main bulk of the specimen was lost. It may be here interesting to chronicle that the remaining portion was inhaled through the nose and mouth; it possessed neither smell nor taste.

The contents of No. 5 were therefore purified and weighed; its density was 2.049.

The contents of No. 1 were also purified by circulation, and had a gravimetric density of 2.245. It lost on circulation a considerable amount of nitrogen which was estimated as ammonia by treatment of the magnesium containing nitride with water. As we are certain that there was no entry of air in preparing the gas, the 34 c.c. of nitrogen must have been evolved from the mineral. It may have been occluded on the surface of the powdered mineral; it need not be remarked that before heating the mineral a nearly perfect vacuum was made in the tube, and that there was no leakage during the operations. We have previously found traces of nitrogen in gas prepared from cleveite; but not all specimens give off that gas. Supposing, however, to take the worst view, the nitrogen had been derived from leakage of air, it would correspond to only 0.3 c.c. of argon.

The contents of jar No. 2 were also purified and weighed. During the purification hardly a trace of nitrogen was removed. The density was 2.209. We have thus:—

Jar No. 1	contains gas of density ..	2.245
" 2	" " ..	2.209
" 6	" " ..	1.979

The light gas which had previously been stored in tubes was now mixed with the light gas from the second set of diffusions, and the mixture was re-diffused fifteen times, involving 90 operations. The density of the lightest portion of this helium was determined by weighing, and found to be 1.988. The helium had, therefore, not been made sensibly lighter by re-diffusion. The mean of the two determinations may be taken as the true density of pure helium; it is 1.98. The refractivity of this sample measured against hydrogen and multiplied by the ratio between hydrogen and air, viz., 0.4564, gives 0.1238. This specimen of light helium of density 1.988 was placed in one of the refractivity tubes, and the lightest helium of the former preparation (density = 1.979) in the other. They had the same refractivity (1000 to 1004). The contents of No. 1, obtained from the mixture of light gases, had the density 2.030, showing that only a little heavier material had been withdrawn.

The lighter fractions of helium were then sealed up in glass reservoirs and stored. The heavier portions were placed in the diffusion apparatus and submitted to methodical diffusion.

After fifteen rounds (ninety operations) the heaviest fraction had density 2.275, the lightest 2.08. The refractivity of the heaviest gas was next determined and

found to be 0.1327. This gas examined in a Plücker's tube showed brilliantly pure helium lines, but along with these the reds and green groups of argon. Calculating from the density of this gas it should contain 1.63 per cent of argon according to the equation $1.961x + 20y = 2.275$. Calculating from the refractivity the percentage of argon should be 1.05, from the equation $1.245x + 0.9596y = 1.33$. A mixture of 99 per cent of the purest helium, and 1 per cent of argon was made, and it showed the argon spectrum with about the same, or with somewhat less intensity than the heaviest gas. Finally, the heavy gas was diffused to the last degree, so that only about 0.5 c.c. remained undiffused; and this small residue, transferred to a Plücker tube, showed the argon spectrum with only a trace of the spectrum of helium. The yellow line and the bright green line were visible, but feeble. This spectrum was compared with that of a mixture of argon with a trace of helium, and nearly the same appearance was to be seen. With the jar in parallel and a spark gap interposed the blue spectrum of argon was equally distinct in both tubes; and, more important still, *there was no trace of any unknown line*. It appears, therefore, that helium contains no unknown gas, nor is it possible to separate it by diffusion into any two kinds of gas; all that can be said is that most minerals which evolve helium on heating also evolve argon in small quantity. This accounts for the difference in density observed in different samples of helium; and in one instance, viz., malacone, the amount of argon evolved on heating the mineral, though small, was much in excess of the helium, so far as could be judged by the spectrum.

In the light of the experiments of which an account has here been given, it is necessary to re-consider the deduction drawn by Professors Runge and Paschen from the complex nature of the spectrum of helium as regards its complex nature. Sir Norman Lockyer has already pronounced in favour of the supposition that helium is a mixture, chiefly on the ground that in the spectra of certain stars some, but not all, of the helium lines are observable. It appears to us that this may well be accounted for by the hypothesis that the differences of temperature and pressure in the stars might produce variations in the spectrum of helium. If a jar and spark gap be interposed while observing the visible spectrum of helium, a profound alteration is to be noticed. The yellow line D_3 is to be seen near the electrodes, and is faint in the capillary portion of the tube, and one of the red lines disappears. The change is not as remarkable as in the case of argon, but is quite distinct and characteristic. Then, as before remarked, the green line becomes relatively stronger at low pressures, so that the light evolved in the tube is no longer the usual brilliant yellow, but dull greenish purple. Is it not likely that the conditions obtaining in the stars may account for the absence of some of the lines ordinarily visible?

If the hypothesis of Runge and Paschen is correct, then the two gases to which they attribute the complex spectrum of helium must have nearly the same density. It has already been shown that by means of the apparatus used for the fractional diffusion of gases it is possible to effect a fair separation of the constituents of air after a few rounds. If the supposed constituents of helium differ in density in as high a proportion as 14 to 16, it is certain that some separation would have been effected. As there has been no such separation, the legitimate inference is that the density of the two supposed constituents does not differ by so great an amount, or that their existence is imaginary. It appears to us that too little is known regarding the nature of the vibrations which cause spectra to make it legitimate to theorise on the subject. It is surely conceivable that an atom may possess such a structure as to render it capable of propagating two different sets of vibrations, each complete in itself, and each resembling the other in general form. Yet it must be acknowledged that our experiments have not disproved the existence of two gases in helium of approximately the

same density; in fact it may be contended that helium is a pair of elements like nickel and cobalt.

We are disappointed in the result of this long research, because we had thought it not improbable that an element of density 10 and atomic weight 20 might prove to be the cause of the fact that different samples of helium possess different densities, according to the mineral from which they are extracted, and also of the separation of helium into portions of different densities by diffusion. We still regard it as by no means improbable that further research will lead to the discovery of the "missing" element, and this appears to be a fitting opportunity of stating our reasons for the belief.

The difference between the atomic weights of helium and argon is $40 - 4 = 36$. Now, there are several cases of such a difference. If we compare the groups of which the first members are fluorine, oxygen, nitrogen, carbon, boron, beryllium, and lithium, we obtain the following table:—

	At. wt.		At. wt.
Fluorine ..	19.0	Boron ..	11.0
Chlorine ..	35.5	Aluminium ..	27.0
Manganese ..	55.0	Scandium ..	44.1
Oxygen ..	16.0	Beryllium ..	9.1
Sulphur ..	32.0	Magnesium ..	24.3
Chromium ..	52.3	Calcium ..	40.1
Nitrogen ..	14.0	Lithium ..	7.0
Phosphorus ..	31.0	Sodium ..	23.0
Vanadium ..	51.4	Potassium ..	39.1
Carbon ..	13.0	Helium ..	4.0
Silicon ..	28.3	?	20.0
Titanium ..	48.1	Argon ..	40.0

The elements helium and argon have been given a provisional place.

The differences between the extreme members of these small groups are given in the short table which follows:—

Manganese—Fluorine	36.0	Chromium—Oxygen ..	36.3
Vanadium—Nitrogen	37.4	Titanium—Carbon ..	36.1
Scandium—Boron ..	33.1	Calcium—Beryllium ..	31.0
Potassium—Lithium	33.1	Argon—Helium ..	36.0

The difference between the atomic weights of argon and helium, it will be seen, is not far removed from those of the other pairs of elements. It appears, therefore, not improbable that there should be an element with atomic weight 20, resembling both argon and helium in its properties. Yet it is not so certain that the middle element should resemble argon and helium, for in the table given it is seen that there are several examples of elements with a middle place which do not resemble those at the extremes. The question is perhaps best left open.

It will be remembered that the gases evolved from a great many minerals and mineral waters have been examined, and that in many cases they have been found to contain helium and argon. In no instance up to the present has any sample of the gases evolved on heating in vacuum been found to show unknown spectrum lines. The amount of argon, as proved by the account which we have just given of our experiments, is very small, and in the case of the gas from cleveite, investigated by Langlet, it is probable that argon was almost completely absent, for it possessed the density 2. In malacone, on the contrary, argon is present in larger amount than helium, although neither gas is obtainable from it in large quantity. It appears to us not beyond the limit of probability that in some as yet uninvestigated mineral the middle member of the helium group may be discovered. When it is considered that germanium, an element which has been recognised only in one of the rarest of minerals, argyrodite, is the middle element of the trio, silicon, germanium, and tin, of which the first and last members are common, it is surely not unreasonable to hope that the middle member of the helium trio may ultimately be found. The amount of helium in fergusonite, one of the

minerals which yields it in fair quantity, is only 33 parts by weight in 100,000 of the mineral, and it is not improbable that some other mineral may contain the missing gas in still more minute proportion. If, however, it is accompanied in its still undiscovered sources by argon and helium, it will probably be a work of extreme difficulty to effect its separation from these gases.

Addendum.—Since this paper was written, Professors Runge and Paschen, in a communication to the British Association in August last, have withdrawn their contention that helium is a mixture, or, perhaps more correctly stated, they now ascribe to helium the same complexity as that of oxygen, the spectrum of which may also be arranged in two series, each consisting of three sets of lines. As oxygen has not yet proved to be complex, the surmise that helium is complex therefore falls to the ground.

FERGUSONITE, AN ENDOTHERMIC MINERAL.*

By WILLIAM RAMSAY Ph.D., LL.D., Sc.D., F.R.S.,

and
MORRIS W. TRAVERS, B.Sc.

THE mineral fergusonite, discovered by Hartwall, occurs in felspar and mica deposits, in the same manner as most of the rare Norwegian minerals, such as euxenite, orthite, samarskite, &c.. The position in which such minerals are found, embedded in masses of felspar, or encrusted with mica, leaves the question of their origin an open one. Whether they are deposited in the felspar by water, or whether they are contemporaneous with the felspar, is a matter of speculation. Fergusonite is a black lustrous mineral, not unlike obsidian in outward appearance, but of considerably higher density. Seen under the microscope, even with the highest power, there is absolutely no sign of crystalline structure, though in thin slices the substance is translucent, and transmits brown-yellow light. It is, however, macrocrystalline, occurring in quadratic sphenoids. It is quite homogeneous, and displays no sign of cavities. Like similar minerals, it contains helium, which is expelled on the application of heat.

But this mineral presents a peculiarity, which has led us to publish this note. When heated to a temperature not exceeding 500° or 600°, it suddenly becomes incandescent, and evolves much of its helium; while its density decreases.

The analysis of the mineral was kindly undertaken by Miss Emily Aston, to whom we desire to express our indebtedness. The mineral has been previously analysed by Hartwell, its discoverer, and by Weber, and, for the sake of comparison, we quote the earlier analyses (Rammelsberg's "Mineralchemie," p. 401):—

Composition of Fergusonite.

	Miss Aston. Hartwell. Weber.		
Oxides of niobium and tantalum	40.95	47.75	48.84
Oxides of yttrium, erbium, &c..	31.09	41.91	38.61
Oxides of cerium, &c..	13.87	4.68	3.05
Uranium dioxide..	3.36	0.95	0.35
Uranium trioxide .	3.81	—	—
	7.17	—	—
Titanium dioxide .	4.56	—	—
ZrO ₂ ..	—	3.02	6.93
Silica ..	1.42	—	—
Ferric oxide ..	1.55	—	—
FeO ..	—	0.31	1.33
Lead oxide ..	0.16	—	—
SnO ₂ ..	—	1.00	0.35
Copper oxide .	0.12	—	—
	100.89	99.62	99.46

The oxides of niobium and tantalum were converted into double fluorides of these metals with potassium fluoride; and on examination of the crystals under the microscope, they were seen to be almost entirely of one form. They were easily soluble in water, and, from previous experience with these compounds, we were able to recognise them as potassium-niobium oxy-fluoride. There appears to be hardly any tantalum-fluoride present in the possible mixture. The uranium dioxide was estimated by heating the mineral with dilute sulphuric acid in a sealed tube, and titrating the dioxide with potassium permanganate. The trioxide was calculated by difference from the total uranium. The cerium metals were separated, as usual, by means of a saturated solution of potassium sulphate.

It is thus seen that fergusonite is mainly a niobate of yttrium, containing oxides of uranium, but in no great quantity.

The gases evolved by the incandescence of nearly 5 grms. (4.852) of the mineral, heated in a vacuum tube, had the following composition:—

	Total gas.	Per grm. of mineral.	Per cent.
	C.c.	C.c.	
Helium ..	5.24	1.080	75.50
Hydrogen ..	0.38	0.078	5.47
Carbon dioxide ..	1.19	0.245	17.14
Nitrogen ..	0.13	0.027	1.88
	6.94	1.430	99.99

The remaining mineral was mixed with hydrogen-potassium sulphate, and heated to redness. More gas was evolved; oxygen, resulting from the decomposition of the sulphuric anhydride, was present in considerable quantity. The sulphur dioxide and the carbonic anhydride were removed by passing the gases through soda-lime, before it entered the pump; hence they do not appear in the analysis.

	Total gas.	Per grm. of mineral.	Per cent.
	C.c.	C.c.	
Helium ..	3.48	0.733	60.3
Nitrogen ..	0.42	0.088	7.3
Oxygen ..	1.87	0.394	32.4
	5.77	1.215	100.0

The mineral taken weighed 4.744 grms.

The density was determined before and after heating. Great care was taken to make sure of the absence of air-bells, by warming the powdered mineral under water in a vacuum before weighing it.

Density before heating ..	5.619
" after " ..	5.375

It is thus seen that the mineral loses density on incandescence.

The amount of heat lost by this curious mineral in parting with its helium was determined. The plan of operation was to burn in oxygen a known weight of hydrogen, ascertained by measuring it, under a small platinum crucible, in a calorimeter. The rise of temperature was noted. This operation was repeated several times, so as to standardise the calorimeter. Some grms. of mineral were then placed in the crucible, and the operation was repeated; the heat evolved by the incandescing mineral added itself to that from the burning hydrogen, and the rise of temperature was greater. Knowing the heat of combustion of hydrogen, a simple calculation gave the heat evolved by the exothermic change in the mineral. The actual data are as follows:—

* A Paper read before the Royal Society, January 20th, 1898.

	I.	II.	III.	IV.
Rise of temperature per gm. of hydrogen ..	14°65'	14°68'	14°47'	14°56'
Additional rise for 6'0595 grms. mineral ..	2'13" = 0°352' per gm.			
Additional rise for 4'0830 grms. mineral ..	1'38" = 0°338' per gm.			
Mean rise per gm. hydrogen ..	14 59°			
Mean rise per gm. mineral ..	0°345'			
Heat of combustion of 1 gm. hydrogen ..	34200 calories.			
Heat of decomposition of 1 gm. mineral ..	809 calories.			

In these experiments a correction was, of course, introduced for the change of temperature of the calorimeter during the experiment, due to the temperature of the surrounding air being higher or lower than that of the calorimeter.

The percentage of helium in the mineral, by weight, is 0'0194, evolved on incandescence, and on further heating, 0'0132; the total percentage is 0'0326.

Dr. Shields was so kind as to determine the specific heat of fergusonite. A Bunsen's calorimeter, in thorough working order, was used. The data are:—

Weight of mineral ..	8'789 grms.
Temperature before introducing into calorimeter ..	17°3° C.
Deflection (1 m.m.=0'001053 K) ..	154'4 m.m.
Mean specific heat between 0° and 17°3'	0'1069

Various questions are raised by the behaviour of this interesting mineral. Its evolution of heat, accompanying its parting with helium, suggests the idea that it is a true of endothermic compound of helium. Had its density, as is the case with alumina, and with other oxides which rise spontaneously in temperature when heated, increased instead of decreasing, the evolution of heat might justly have been ascribed to polymerisation. But an evolution of heat, accompanied by a fall in density, leads to the conjecture that the loss of energy is the result of the loss of helium; and that, conversely, the formation of the compound must have been concurrent with a gain of energy. That the helium is actually in combination, and not retained in pores in the mineral, is evinced by there being no pores in which the helium might be imprisoned. Surface-absorption is equally out of the question, for the mineral is compact. The only remaining possibility is that the helium is in chemical combination. And if this is true, then the compound must be an endothermic one.

The question next arises, with what constituent of the mineral is the helium in combination? This question cannot at present be answered. All that can be said is that the amount of helium does not appear to depend on the total percentage of uranium, although minerals containing uranium usually (probably always) contain this element. Even in English pitchblende there was found a trace of helium. And in malacone, a mineral containing no uranium, a trace of helium was found; also in a specimen of meteoric iron. The presence of niobic and tantalic anhydrides, and of the yttrium group of elements, is also favourable to its presence. But the proportion between the weight of the helium and that of the other elements present makes any calculation of the atomic relations between the helium and the other elements out of the question.

There is one other substance at least which decreases in density while it evolves heat; that substance is water, in changing into ice. The effect of compressing ice is to lower its melting-point, and at the same time to reduce its heat of fusion. At a sufficiently high pressure there would be a continuous transition from ice to water, no heat change taking place during the transition. Matters

would be in a similar condition to those which accompany the change of a liquid into gas at the critical temperature; the smallest alteration of temperature would be enough to bring about the change. In speculating on the origin of such a remarkable compound, is it not allowable to guess that it represents a condition of our earth realised only before solidification had set in? That these minerals, containing the rare elements, represent a portion of the interior of our planet; and that, under the enormous pressure obtaining at the centre, combination with helium was an exothermic event; and that such compounds, having by some unexplained accident come to the surface of the globe, where they are no longer exposed to such pressure, they have, in consequence of the change, become endothermic? The frequency of the helium spectrum in the stars, and its presence in the sun, makes it less improbable that some such explanation may lie not far from the truth.

There are at least two other minerals, gadolinite and æschinite, which exhibit endothermic properties. But these minerals, instead of decreasing in density on ignition, increase. The following table shows the gases evolved when they are heated, their densities before and after heating, and the loss of weight which they suffer:—

	Gases evolved. C.c. per gm.				Density.		Loss of weight.
	H ₂ .	CO.	CO ₂ .	He.	Before.	After.	
Gadolinite	0'700	0'011	1'060	none	4'289	4'371	0'82
Æschinite	0'458	none	0'215	0'243	4'685	4'793	1'018

It is to be noticed that only the æschinite contains helium, and that in very small quantity.

The fact that these minerals increase in density, and that only one yields helium, places them in a different class from fergusonite. Moreover, the rise of temperature is not to be compared to that seen with fergusonite, for the glow is barely visible.

ON THE PREPARATION AND PROPERTIES OF PERCARBONATE OF POTASSIUM.

By A. VON HANSEN.

In a memoir I recently published in collaboration with M. E. J. Constam (*Monsieur Scientifique*, August, 1897) we indicated the method of preparing percarbonate of potassium, as well as the general reactions of this new body. I have undertaken fresh researches, with the object of determining the conditions most favourable to its production, and to study some of its particular properties more closely.

As we have previously stated, percarbonate of potassium is obtained by the electrolysis of a concentrated solution of carbonate of potash maintained at a low temperature. The solution at the anode, clear at first, gradually assumes a milky appearance, because of the separation of a sky-blue solid body, viz., percarbonate of potassium. The product, however, is not free from ordinary carbonate. It is therefore indispensable to find conditions of temperature, concentration, and density of current, which will allow of the highest yield of percarbonate of potassium.

My first experiments were made with rather primitive apparatus, as previously described. A porous pot of 200 c.c. capacity was placed in a glass beaker of such capacity that the free space for the anode between the two receptacles was about 120 c.c. As anodes I made use of platinum spirals, and as cathodes sheets of nickel.

To follow the progress of the electrolysis, it was essential to have a method of titrating by which at any given moment the proportion of percarbonate present could be determined. The method we first adopted consisted of

pouring the solution of percarbonate into an excess of a solution of Mohr's salt, and to titrate the excess of unoxidised iron with permanganate of potash; but this was abandoned as giving doubtful results. I subsequently noticed that percarbonate of potassium treated with dilute sulphuric acid decomposes quantitatively into sulphate of potassium, carbonic acid, and peroxide of hydrogen. This latter can be easily titrated with permanganate of potash.

In all my experiments I have therefore thus determined the proportion of percarbonate of potassium in the anode solution at regular intervals of time. As for the density of the current, that was determined by Oettel's method.

a. Influence of Temperature.

If we commence the electrolysis at -15° we notice that slight variations of temperature have no appreciable effect on the return, if the solution at the anode is very concentrated. The temperature has even been raised to 0° without the reaction being diminished. On the other hand, with solutions of carbonate of potassium of a density below 1.52, the useful return by the current diminishes as the temperature rises. It therefore follows that towards the end of the operation, when the concentration has been diminished by the separation of the percarbonate of potassium, the temperature ought to be lowered.

b. Influence of Concentration.

If the concentration of the liquid at the anode varies ever so little from saturation, we obtain but little, if any, percarbonate in the solid state, on account of the great solubility of this latter body in dilute solutions of carbonate of potassium. As the concentration diminishes, we notice a decrease in the useful effect of the current,—not so much because of the decomposition of the percarbonate already formed, as by reason of the formation of bicarbonate of potash. This latter is precipitated in large quantities in the crystalline state.

c. Influence of the Density of the Current.

To obtain a product rich in percarbonate of potassium it is essential that the density of the current should be very high. A current of low density favours the formation of bicarbonate of potassium. In a series of experiments, where the density of the current varied from 0.3 to 2 ampères per square decimetre, the proportion of percarbonate produced varied from 25 to 55 per cent. Each experiment was carried out on a solution of carbonate of potassium of 1.56 density, and the temperature was kept constantly at -10° . By increasing the density of the current to 30 and 60 ampères per square decimetre, the proportions of percarbonate of potassium produced, rose to 85 and 95 per cent. In all these experiments, then, it appears that—everything else being equal—a good return of percarbonate of potassium is correlative with a high current density, and a concentration as near saturation as possible. It is not absolutely necessary to maintain the temperature at -12° , if we only take care to keep the liquid at the anode as near saturation as possible.

The density of solutions of pure carbonate of potassium, saturated at the ordinary temperature, is from 1.555 to 1.565. It is curious to note that in lowering the temperature of these saturated solutions there is no separation of any solid salt, although such a separation takes place with ease if we use solutions of commercial carbonate of potassium, even when the temperature is lowered only a few degrees below zero.

To follow the progress of the reaction I took, at regular intervals, a known volume of the anode solution, and titrated the percarbonate of potassium. Although the liquid was carefully stirred before taking each sample, it is a matter of some difficulty to obtain a sample representing the average contents of the apparatus. To do the titration, 1 c.c. of the anode solution is poured into

150 c.c. of dilute sulphuric acid, and the peroxide of hydrogen formed is titrated with permanganate of potash. Among the numerous experiments I have made in this manner I will pick out a few, of which I now give the details.

I.

Density of the anode solution: 1.550.

Volume of the anode solution: 55 c.c.

Density of the current: 40 ampères per square decimetre.

Am- pères.	Tempera- Volta. ture. °C.	Time in minutes.	Solution of KMnO ₄ . C.c.	Per- carbonate. C.c.	Percarbonate per ampère-hour.
2.5	1	15	2.14	0.0375	3.1
2.5	7.3	30	4.74	0.0850	3.4
2.5	10	45	7.15	0.1199	3.3
2.5	10	60	9.18	0.1609	3.3
2.5	7.6	75	12.09	0.2103	3.4
2.5	10	90	13.30	0.2331	3.1

At the commencement of the operation the useful return from the current is thus fairly constant; but if we prolong the experiment it decreases rapidly, on account of the diminution of the concentration: this diminution is due to the formation of bicarbonate of potassium.

II.

Density of the anode solution: 1.560.

Volume of the anode solution: 150 c.c.

The anode was formed of a platinum wire 48 c.m. in length and 0.0345 c.m. diameter; the total surface was 5.2 square centimetres; current intensity = 3 ampères; difference of potential at the electrodes = 8.1 volts.

We obtained 19.5 grms. of the salt, containing 92 per cent of pure percarbonate of potassium. Duration of the experiment, two hours and thirty minutes. Total quantity of electricity, 7.5 ampère-hours. Return of percarbonate per ampère-hour, 2.4 grms.

At the termination of the experiment titration showed 21.84 grms. of percarbonate in the total volume of the solution, or 2.9 grms. per ampère-hour. The filtered liquid was also titrated; it contained 3.74 grms. of percarbonate of potassium.

At the end of the operation the density of the anode solution was 1.42. During the entire time of the experiment the temperature varied from -12° to -10° . As a rule, at the beginning of the electrolysis, we can detect the characteristic odour of ozone, but this gradually disappears as the operation proceeds.

As cathode solution we use a less concentrated solution of carbonate of potassium, so as to prevent the salt crystallising out. As anode, the only convenient metal is platinum. Iron, nickel, and copper are all attacked very rapidly. An aluminium vessel serves very well to hold the refrigerating materials. With 8 kilogrms. of a mixture of ice and sea-salt we can keep the temperature at -15° for three hours.

The process I have just described is not very practicable when we want to produce large quantities of percarbonate of potassium, both because of the diminution of the concentration which goes on and the lowering of the useful effect of the current which is the natural consequence.

For preparing the percarbonate on the large scale we make use of an apparatus provided with an appliance by means of which we can introduce fresh portions of potassic carbonate solution into the lower part of the positive compartment, as the percarbonate is precipitated. As the density of the fresh solution is greater than that of the electrolysed solution, the latter will be displaced mechanically, and will run off through an orifice provided in the upper part of the jar. By taking certain precautions we can maintain the operation continuously for a considerable number of hours.

The precipitated percarbonate is filtered on a filter-pump; it contains 87 to 93 per cent of pure percarbonate of potassium. It is then spread out on porous plates,

and placed in an apparatus traversed by a current of dry air : by this means it can be dried very rapidly, and the resulting salt does not contain more than 2 to 4·3 per cent of water. By first drying the air over concentrated sulphuric acid and melted chloride of lime, and prolonging its action for twelve hours, we can further reduce the proportion of moisture present to 0·6 or 0·8 per cent. Towards the end of the drying the temperature of the air may be raised to 40°. Above this temperature decomposition commences. By heating the air to 100° the proportion of percarbonate present drops by 6 per cent at the end of an hour, and by 10 per cent after two hours.

Percarbonate of potassium thus obtained is an amorphous powder of a slightly blue tint. The blue colouration of the wet salt almost completely disappears on drying.

Percarbonate of potassium decomposes easily, giving up its oxygen to oxidisable bodies. Its instability, however, is not so great as would have been at first thought ; to decompose it rapidly it is necessary to submit it to a temperature of 200° to 300°. Immersed in water it only decomposes slowly. If, for example, we place 0·3 grm. of percarbonate of potassium into 50 c.c. of water, and then titrate with permanganate of potash, after adding dilute sulphuric acid, after the lapse of half an hour, we find that the loss is hardly 1 per cent. But at a higher temperature the splitting up into oxygen and bicarbonate of potassium takes place very rapidly. Thus percarbonate of potassium constitutes a primary body most convenient for the preparation of oxygen in laboratories. After having put the salt in water, it is only necessary to raise the temperature to 45° to obtain a very regular flow of gas. The decomposition is complete without having to raise the temperature any higher than 45°, and we finally obtain a solution not containing any percarbonate at all. From 100 grms. of the salt we can obtain about 5 litres of oxygen. The traces of carbonic acid which might be given off at the same time are absorbed by an alkaline solution.

We have seen that, under the influence of dilute acid, percarbonate of potassium decomposes with the formation of peroxide of hydrogen ; here is at once a simple method for rapidly preparing solutions of peroxide of hydrogen, and this is the more important as commercial peroxide of hydrogen changes very rapidly, whereas percarbonate of potassium keeps very well indeed if it has been carefully dried. It suffices to keep it in hermetically sealed glass flasks.

The percarbonate is but very slightly soluble in alcohol, and it has up to the present time been quite impossible to obtain it in crystals from its aqueous solution. If we saturate water at 0° with percarbonate of potassium, and then lower the temperature to -15°, we obtain a voluminous crop of crystals, but they are formed almost entirely of ice.

It is possible to enrich impure percarbonate of potassium by a very simple process. Into a moderately concentrated solution of caustic potash we place an excess of the percarbonate to be purified, and leave it for several hours at a temperature of -8° or -10°. Under these conditions the bicarbonate held by the percarbonate, is decomposed by the caustic potash with the formation of a neutral carbonate which goes into solution. By filtration we obtain a percarbonate containing a very small quantity of caustic potash, which can be extracted by treating it with absolute alcohol. The final product then contains about 95 to 99 per cent of percarbonate of potassium.

I applied this system of purification to a percarbonate which titrated 78 per cent. Analysis gave me the following results :—

Substance used	0·5012 grm.
Carbonate of potassium	0·3912 "
Carbonic acid	0·1241 "
Water	0·0025 "
Oxygen	0·0434 "

The salt was placed in a porcelain boat in a tube of Bohemian glass, and the whole was warmed. The carbonic acid and the water were estimated in the ordinary way ; the residue of carbonate of potassium was weighed with great care. The figures worked out into percentage give for the composition of the purified salt :—

Percarbonate of potassium ..	95·68 per cent
Bicarbonate of potassium ..	3·84 "
Carbonate of potassium ..	0·35 "
Moisture	0·10 "

99·97 "

I have not yet been able to isolate the percarbonates of sodium or ammonium. But I am carrying on a new research in that direction.—*Zeitschrift für Electrochemie*, 1897, p. 455.

ON THE SEPARATION AND ESTIMATION OF IODINE, BROMINE, AND CHLORINE.

By AD. CARNOT.

MANY methods have been proposed for the estimation of these three bodies in a mixture of haloid salts. In deciding to offer still another one, it is because it appears to unite the qualities of simplicity, rapidity, and exactness, which are not found altogether in any of the other already known methods.

I have made use of the excellent instructions given by Fresenius (Fresenius, *Chim. Quantit.*, sixth French edition, p. 406) for the estimation of the iodine, and I have profited by some observations of M. Dechan (*Chimie Soc.*, vol. xlix., p. 682 ; *Bull. Soc. Chim.*, vol. ii., p. 331, 1887), and of M. Baubigny (*Comptes Rendus*, vol. cxxv., November 2, 1897, p. 654) relative to bromine.

The new method is founded on the following reactions :—In a mixture of chlorides, bromides, and iodides in solution, sulphuric acid charged with nitrous vapours is able to entirely displace the iodine in the cold without acting on the hydrochloric or hydrobromic acids in any way ; the iodine can then be entirely dissolved and removed by sulphide of carbon. By adding sulphuric and chromic acids the bromine can only be partially removed in the cold, but by heating to about 100° for about half an hour or an hour, and then letting it cool, and dissolving in sulphide of carbon, it can be entirely removed. In neither case is there a trace of chlorine set at liberty, the estimation of this body can then be made with nitrate of silver.

As for the iodine, it is estimated volumetrically by adding hyposulphite of sodium to the sulphide of carbon until the decolouration is complete ; for the bromine we operate in the same manner, but first add a little iodide of potassium, then adding titrated hyposulphite of sodium until the violet colouration, caused by the free iodine exactly disappears.

A series of experiments made on most varying proportions of chlorides, bromides, and iodides, have proved the accuracy of the results, provided that the precautions about to be mentioned were duly observed.

1. *Iodine*.—The neutral solution of the salts, diluted to about 200 c.c. in volume, is introduced into a bulb-shaped funnel of 350 c.c. to 400 c.c. capacity, well closed at the top with a ground stopper, and at the lower part by a glass tap, made sufficiently strong as not to run any risk of breaking when heated in the water bath.

Into the cold solution we drop about ten drops of sulphuric acid saturated with nitrous fumes (produced by the action of concentrated nitric acid on starch), we then pour in from 10 to 15 c.c. of pure sulphide of carbon. The glass stopper is then fixed firmly in, and the whole shaken vigorously several times ; the sulphide of carbon is now allowed to collect, shaking gently to help the small drops

Proportions taken for the experiment.						Hyposulphite used for		Weighted.	Found.		
Iodide.	Bromide.	Chloride.	I.	Br.	Cl.	I.	Br.	AgCl.	I.	Br.	Cl.
M.grms.	M.grms.	M.grms.	M.grms.	M.grms.	M.grms.	C.c.	C.c.	M.grms.	M.grms.	M.grms.	M.grms.
200	100	200	153.0	67.2	95.2	22.3	15.2	380	152.5	65.3	94.0
100	100	100	76.5	67.2	47.6	11.2	15.4	—	76.6	66.2	—
100	200	100	76.5	134.4	47.6	11.2	31.0	192	76.6	133.3	47.5
20	40	200	15.3	26.9	95.2	2.2	6.3	—	15.0	27.1	—
5	50	200	3.8	33.6	95.2	0.6	7.8	—	4.1	33.5	—
1	100	600	0.8	67.2	285.6	0.1	15.3	—	0.7	65.8	—
—	20	500	—	13.4	238.0	—	3.1	—	—	13.3	—
—	10	200	—	6.7	95.2	—	1.5	—	—	6.4	—
—	5	200	—	3.4	95.2	—	0.7	—	—	3.0	—
—	1	200	—	0.7	95.2	—	0.1	—	—	0.4	—
100	100	20	76.5	67.2	9.5	11.2	15.5	39	76.6	66.6	9.6
100	100	5	76.5	67.2	2.4	11.2	15.6	10	76.6	67.1	2.5

which adhere to the sides of the glass to become detached and sink to the bottom. The sulphide of carbon is of a deep violet colour if much iodine is present, and pale violet, or even rose colour, if there is only a little; it is easily distinguished in the aqueous solution, and it fills the lower part of the bulb and the fine tube down to the tap. The tap is now carefully opened, and the coloured sulphide of carbon run on to a filter-paper moistened with water; the tap is shut the moment the aqueous solution reaches it. Another 10 c.c. of sulphide of carbon is added, and the shaking repeated as before; the reagent is, as a rule, but very faintly coloured with iodine the second time; three or four more drops of nitrous sulphuric acid are then added, and after a further agitation, which ought not to produce any change of colour if the first operation was properly carried out, the sulphide of carbon is allowed to collect, and run off into the same funnel, which must be covered over by a sheet of glass to prevent evaporation.

The introduction of two or three c.c. of sulphide of carbon, and one or two drops of nitrous sulphuric acid, enables us to collect the small drops of sulphide which may remain on the surface of the liquid, to make sure that the displacement of the iodine is complete, and to wash out the small quantity of faintly coloured sulphide contained in the passage through the glass plug of the tap.

The sulphide of carbon, collected on a moistened filter, is well washed with cold water. Only the first washings are retained, and added to the aqueous solution in the bulb for the continuation of the analysis. By piercing the filter we can run the sulphide of carbon into a small glass-stoppered flask containing about 30 c.c. of a $\frac{1}{2}$ per cent solution of bicarbonate of sodium. Then by means of a small graduated burette we run in a titrated (decinormal or centinormal) solution of hyposulphite of sodium, until the sulphide of carbon is completely decolourised. The flask must be well shaken after each addition of the reducing agent; the reaction is most distinct, and the exactness of the results is as close as possible, not only in the presence of chlorides, as was observed by Fresenius, but also in the presence of bromides.

II. Bromine.—To estimate the bromine we pour a few cubic centimetres of 10 per cent chromic acid and three or four cubic centimetres of sulphuric acid, containing its own volume of water, into the large bulb; we then replace the glass stopper, taking care to fix it in very firmly. The bulb is then floated in a water bath at 100° for from half an hour to an hour; it is then taken out and allowed to become completely cold; sulphide of carbon is then introduced, and the operation proceeded with, as I have already explained for iodine, by three successive exhaustions. The sulphide of carbon is collected on a moistened filter, then washed with cold water until it is no longer acid. It is then run into a glass-stoppered bottle, where a little solution of iodide of potassium, and 30 cubic centimetres of bicarbonate of sodium is added to it. The bottle must be well shaken several times. The bromine displaces an

equivalent proportion of iodide which, being set free, gives the solvent a violet colour which is much more easily visible than was the yellow tint produced by the bromine. The estimation of the iodine is made, as before, by titrated hyposulphite of sodium, and we have then only to multi-

ply the weight of the iodine by the coefficient $\frac{80}{127}$ to get the weight of the bromine present.

III. Chlorine.—The acid solution from which the iodine and the bromine have been removed, and to which both the first washings have been added, is poured into a large beaker and diluted to about 500 c.c. Nitrate of silver is then added, and the whole heated to collect the chloride. The precipitate will be found to be slightly coloured by chromate of silver; to purify it the liquor is decanted after cooling, and replaced by a little warm water slightly acidulated with nitric acid; it is again allowed to cool, and the precipitate is then washed by decantation. The chloride of silver, now perfectly white, is dried and weighed on a tared filter with the usual precautions.

Of the three, the estimation of the chlorine is the one to which, as a rule, we attach the least importance. Instead of operating in the manner I have just described, we might nearly always be satisfied to estimate by difference; a volumetric method enables us to do this rapidly in the following manner.

We can operate on the solution deprived of iodine by dividing it into two portions. One is treated for the estimation of bromine only, as has been described; the other is precipitated by a measured quantity of titrated nitrate of silver. The excess of silver is then determined by means of titrated sulphocyanide, using iron-alum as an indicator. According to the estimation of the bromine, we know the quantity of nitrate of silver it has used, and, by difference, we can easily calculate the chlorine. Thus we need (and, above all, if the chlorides are present in much greater proportion than the iodides and bromides) only take one-tenth or one-fiftieth of the first neutral solution, add to it chromate of potassium as an indicator, and by means of a burette drop in titrated nitrate of silver until the appearance of the red colour of the chromate. Thus, taking from the nitrate of silver used the quantity which corresponds to the iodine and the bromine, we get what was precipitated by the chlorine.

The chlorine, it is seen, can be easily estimated by either of these methods.

The accompanying table, showing the results of a series of experiments made on both large and small proportions of each of these three bodies, enables us to appreciate the exactness which can be habitually attained by this method.

For a simple *qualitative* research for small quantities of iodine and bromine in the presence of a large excess of alkaline chlorides, the following plan is to be recommended:—

1. Separate the iodine in a small quantity of the neutral solution by means of nitrous sulphuric acid, and collect it in a few drops of sulphide of carbon. The violet or rose colouration is extremely sensitive.

2. The iodine having been eliminated, add to the solution, in a small flask, a small quantity of chromic and sulphuric acid, heat to boiling, and place over the opening a strip of yellow fluoresceine paper, described by M. Baubigny, both with regard to preparation and sensitiveness to bromine. The faintest traces of bromine will be shown by the characteristic rose colour. — *Comptes Rendus*, cxxvi., No. 3.

THE REVIVAL OF ALCHEMY IN FRANCE.*

By H. CARRINGTON BOLTON, Ph.D.

RECENT discoveries in physics, chemistry, and psychology have given disciples of Hermes renewed hopes, and the present position of chemical philosophy has given the fundamental doctrine of alchemy a substantial impetus; the favourite theory of a *prima materia*, or primary matter, the basis of all the elementary bodies, has received new support by the discoveries of allotropism of the elements, isomerism of organic compounds, the revelations of the spectroscopic, the practical demonstrations by Norman Lockyer, the experiments on the specific heat of gaseous bodies at a high temperature by Mallard and Le Châtelier, the discoveries of Sir William Crookes, as set forth in his monograph on "Meta-elements," the discovery by Carey Lea of several singular allotropic forms of silver, and, most weighty of all, the mass of related facts and phenomena which find their ultimate expression in the Periodic Law of the Elements, so that many chemists of the present day are inclined to believe in the mutual convertibility of elements having similar chemical properties. Daniel Berthelot, in his notable work entitled "De L'Allotropie des Corps Simples," boldly affirms his belief in the unity of matter. He says: "Without seeking to find in any one of the known elements the generator of the others, can we not invoke the facts that we have revealed in our study of carbon in favour of the hypothesis of a unique matter unequally condensed?" And elsewhere he writes: "The transmutation of an element is nothing more than the transformation of the motions which determine the existence of said element, and which gives it special properties into the specific motions peculiar to the existence of another element."

Simultaneously with the development of the truly scientific aspect of alchemical theory, there has arisen an extraordinary revival of the metaphysical side of the question; this goes hand in hand with the interest in chiromancy, astrology, theosophy, and occult sciences which occupy so large a place in modern thought, literature, and polite society on both sides of the Atlantic. This tendency to cultivate the esoteric manifests itself in the study of the Kabala, the investigation of the mysteries of Buddhism, Confucianism, and other oriental philosophies, in researches into the phenomena of spiritualism so-called, and in the foundation of societies to study Psychic Force and the tenets of the followers of Madame Blavatsky; crystal-gazing, reading in magic mirrors, slate-writing, planchette, the quasi-scientific study of apparitions, of table-turnings, of rappings by unseen powers, of telepathy, of subliminal self, are now regarded as legitimate pursuits in no wise necessarily associated with the black arts of mediæval times, provided only they are conducted in a spirit of enquiry, and for the purpose of discovering the latent power underlying these phenomena. And this line of research receives stimulus from the results secured by students of experimental psychology, of hypnotism, from such discoveries as the phenomena of the X-rays, and from the transcendental physicists who theorise on the miraculous consequences of four dimensional matter. Crowded lecture halls reward exhibitions of trance mediums, speakers on theosophy, palmistry, and occultism; in lower walks of life fortune-tellers and clair-

voyants reap a modest harvest; books treating of occult themes enjoy great notoriety; writers of fiction find it profitable to introduce the mysterious into the children of their brains; even secular journals, especially those of France, give space to the all-absorbing discussions on hermetism; these are some of the evidences of great popular interest in the unknowable. Only persons with special intellectual equipment are able to measure, weigh, sift, and co-ordinate the novel phenomena gathered by researches in the field of hypnotism, psychology, and occultism; those of weaker mental powers fail to perceive the real significance of the discoveries, and are led away into unprofitable and dangerous superstitions.

In the Middle Ages alchemy was nurtured by ignorant superstition; now it is fostered by the prevalent devotion to esoteric studies; formerly the popular belief was in part supported by the fraudulent claims of impostors; now a higher standard of intelligence rejects the transparent tricks of imitators of Cagliostro. It is not by sleight-of-hand that the revival of alchemy is now being engineered, but by a company of educated charlatans.

The movement to reascitate alchemical doctrines and practices has been particularly successful in France, where there are to-day four societies and a "university" claiming to possess occult knowledge of hermetic mysteries. These secret societies are named, "Ordre de la Rose-Croix," "L'Ordre Martiniste," "La Société d'Homéopathie Hermétique," and "L'Association Alchimique de France."

The first two of these societies seem to work on lines similar to Free Masonry, and claim that their secret mysteries were bequeathed by the last sages of Atlantis, and by the Lemures to their brethren in Asia and Egypt, dwellers in sanctuaries whence issued Krishna, Zoroaster, Hermes, Moses, Pythagoras, and Plato. The priestly Magi who preserved this lore in the temples of Thebes, Heracleopolis, Aphrodite, Pthah, and Serapis, were succeeded by secret alchemical societies of the first centuries of our era; then followed the Hermetic lodges of the Arabs, and these gave rise to the Templars, the Rosicrucians, and the Martinists.

The third society cultivates especially occult therapeutics, a system of medicine invented in the sixties by Count Cæsar Mattei, of Bologna, which unites the principles of Hahnemann with those of the Iatro-chemists, disciples of Paracelsus. This new departure in medicine publishes four monthly organs, and special treatises all its own.

The Alchemical Association of France is successor to the Société Hermétique, which was founded by the late Albert Poisson (†1894), also known by the pen-name "Philophotes." Its seat is in Paris. The objects of the Association, as set forth in its constitution, are "the theoretical and experimental study of evolution, and of the transmutation of bodies. Its members, with this end in view, study the processes of the ancient alchemists, and compare them with the work of modern chemists." These objects are to be accomplished as follows: "The Association proposes to assist in reviving the unitary doctrines of chemistry: 1st, by grouping the efforts of isolated workers by means of *L'Hyperchimie*; 2nd, by furnishing them the aid of advanced students; 3rd, by supplying, so far as possible, books and apparatus to its members. Researches of the members, when approved by the masters, should be forwarded in duplicate to the secretary-general; one will be printed in *L'Hyperchimie*, and the other will be preserved in the archives of the Association for the benefit of members who can secure it on demand." "Candidates for admission must pass an examination in: 1st, the theory and history of alchemy; and 2nd, the elements of physics and of chemistry (without mathematics). A diploma from a normal, polytechnic, or industrial school will be accepted in place of No. 2."

The affairs of the Association are controlled by the secretary-general, F. Jollivet-Castelot (of Douai), assisted

* Abstract of a Paper read to New York Section of the American Chemical Society, October 1, 1897. From *Science*, N.S., vol. vi., No. 154.

by seven councillors, who hold an annual meeting. There are at present (July, 1897) two honorary members, Camille Flammarion, the popular writer on astronomy, and August Strindberg, a Swede residing in Austria, author of several hermetic essays.* There are two other classes of members, masters (*maîtres*), who are chosen from the ordinary members by the Council after an examination of their writings, and ordinary members (*membres adhérents*), of which the number is unlimited. Modest dues entitle the members to the organ of the Association, *L'Hyperchimie*, a monthly review of alchemy and hermetism founded in 1896.

The councillors of the Alchemical Association have combined with the active members of the other societies named to establish a *Université Libre des Hautes Etudes*. At present this includes three faculties:—

I. *Faculté des Sciences Hermétiques*, of which the Association Alchimique forms a section. The director of this faculty is Dr. G. Encausse, and the course of instruction embraces the study of the *Tarot*, alchemical philosophy and practice, occultism, mysticism, Hebrew, &c.; the curriculum leading to the "baccalauréat-en-Kabbale" is under the supervision of the group of esoteric students, while candidates for the degrees of master and doctor are under the direction of the Martinist Order.

II. *Faculté des Sciences Magnétiques*, represented by the Ecole de Magnétisme de Paris, and under the direction of M. Durville; it has branches at Lyons, Bordeaux, and other cities.

III. *Faculté Spirite*, comprising several sections of spiritism.

Each faculty preserves complete independence, being "united only by moral bonds destined to hasten expansion of the rational spiritualistic movement."

The nature of the instruction given at this University will appear in the review of the philosophy of its promoters.

One of the oldest workers in the Alchemical Association is the "Master" Théodore Tiffereau. In 1854-55 he sent to the French Academy of Sciences six memoirs in which he claimed to have discovered a method of converting silver into gold. Tiffereau had made his experiments in Mexico at great expense, supporting himself meanwhile by taking daguerreotypes. His process was repeated at the Mint, in Paris, before the assayer, M. Levot, but with little success. The substance of his memoirs was published in 1855 in a volume, entitled "*Les Métaux sont des Corps Composés*." Of this a new edition was published by Lermina in 1889. Tiffereau has never abandoned his claim, and as recently as October, 1896, he addressed another memoir to the Academy, in which he attempts to prove that the metal aluminum is a compound. Briefly stated his process is as follows: He placed in a stout glass tube a piece of aluminum foil with pure nitric acid, and sealed the tube hermetically. He then exposed the tube and contents to the sun's rays during two months; at the end of this time he opened the tube; it gave out an odour which he thought was due to ether, and it yielded a few grms. of crystals which he thought tasted like acetic acid. Since both ether and acetic acid are compounds of carbon, Tiffereau concluded that this element was derived from the aluminum. Analytical chemists would criticise this experiment in several points; they would say Tiffereau did not demonstrate the absence of carbon in the metal used, and that he depended upon smell and taste for proofs of the carbon compounds; the tongue and the nose are incontestably useful adjuncts to the reagents of a chemical laboratory, but additional tests for ether and acetic acid would have been more conclusive. In Tiffereau's recent writings he attributes the transmutation of a base metal into the most precious one, to the action of the "microbe of gold."

(To be continued).

* Dr. Stephen H. Emmens, of New York, has been added to the list of honorary members since writing the above.

NOTICES OF BOOKS.

The Early History of Chlorine. Alembic Club Reprints, No. 13. Edinburgh: Wm. F. Clay. London: Simpkin, Marshall, Hamilton, Kent, and Co., Lim. 1897. Pp. 48. No. 13 of these interesting reprints consists of papers by Scheele (1774), Berthollet (1785), Guyton de Morveau (1787), and Gay-Lussac and Thenard (1809), and we are informed in the preface that the volume containing Davy's researches (No. 9) may be taken as a continuation of the present one.

The first paper, by Scheele, contains the account of his accidental discovery of chlorine: he noticed that when pure *spiritus salis* was poured on to finely-ground manganese, and let stand an hour in the cold, the solution became brown, and on warming gave off an odour like warm *aqua regia*; after a quarter of an hour both colour and smell had gone. He then goes on to describe the action of *marine acid* on manganese, and the phenomenon which occurs proves, he thinks, the presence of phlogiston.

In the next paper, by Berthollet, on "Dephlogisticated Marine Acid," the author still adheres to the idea of phlogiston, and he elaborates a new but erroneous theory that chlorine is the higher oxide of an unknown radical. By the time we get to Gay-Lussac and Thenard, ideas have changed, phlogiston is abandoned, and it is sometimes claimed that it was these authors who first established the elementary character of chlorine. It is of the greatest interest to notice the manner in which these early chemists were, so to speak, groping about in the dark, and with what untiring patience they laid the foundations for our modern accepted theories; who knows but that in another hundred years, or even less, we ourselves may not be proved to be as mistaken in many of our present theories as we think they were.

Researches on Molecular Asymmetry. By LOUIS PASTEUR (1860). *Alembic Club Reprints, No. 14.* Edinburgh: Wm. F. Clay. London: Simpkin, Marshall, Hamilton, Kent, and Co., Lim. 1897. Pp. 46.

THE two lectures which comprise this volume were published by the Chemical Society of Paris in 1861.

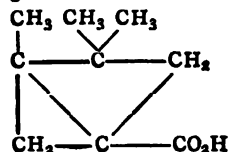
The author begins by reviewing the optical work done by Malus, Huygens, Newton, Arago, and Biot, especially on polarised light; he then goes on to discuss hemihedry in quartz, and then in crystals of organic substances. His own work on this subject, which forms the greater part of the book, is, however, so well known, that we need not go fully into it here; his remarkable discovery of the right- and left-handed tartrates, and the work he did on them, holds as good now as it did when first printed thirty-seven years ago, the whole forming a most interesting volume.

CORRESPONDENCE.

CAMPHOR AND ITS DERIVATIVES.

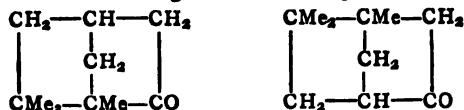
To the Editor of the Chemical News.

SIR,—In the recent discussion of camphor and its derivatives in the CHEMICAL NEWS two or three formulæ are proposed which appear to me inconsistent with established facts. Professor Perkin (CHEMICAL NEWS, vol. lxxvi., p. 287) gives the following formula for isolauronic acid—



This formula can scarcely be reconciled with the fact that the acid conducts itself as an unsaturated acid, reducing instantly an ice-cold neutral solution of potassium permanganate. It has also been shown (*Ber. d. Chem. Ges.*, xxix., 2327) that the acid readily takes up two atoms of hydrogen, on treatment with amyl alcohol and sodium. The resulting saturated cyclic acid gives an α -brom-derivative, and this, in turn gives the original isolauronic acid when warmed with alcoholic potash. (In that paper the acid is called *cis*-campholytic acid, for reasons given in the *Am. Chem. Journ.*, xvii., 430). A reasonable explanation of these relations on the basis of Professor Perkin's formula appears to be impossible.

In the *CHEMICAL NEWS* (vol. lxxvi., p. 297) Dr. Forster discusses the following formulae for camphor:—



He prefers the second formula. Of the two, however, the following considerations appear to prove that only the first could be true. The iso-nitroso-camphor of Claisen and Manasse gives α -camphoraminc acid on treatment with hydrochloric acid (*Ann. Chem.*, Liebig, cclxiv., 80); this in turn gives aminolauroic acid by Hofmann's reaction (*Am. Chem. Journ.*, xvi., 506). β -Camphoraminc acid gives, by the same treatment, dihydroaminocampholytic acid. The relative rates of esterification of aminolauroic and dihydroaminocampholytic acids proves that, if only one of these acids contains a tertiary carboxyl-group, that group must belong to the aminolauroic acid (*Am. Chem. Journ.*, xviii., 686). But since, from the relation of that acid to isonitroso-camphor, its carboxyl must correspond to the carboxyl group of camphor, it follows that in the latter the carboxyl group is combined with a carbon atom of the nucleus, which is not combined directly with hydrogen.—I am, &c.,

W. A. NOYES.

Rose Polytechnic Institute,
Terre Haute, Indiana, January 8, 1898.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 3, January 17, 1898.

M. Schiaparelli, correspondent of the Astronomical Section, writes to deny the announcement of his death.

The Separation and Estimation of Iodine, Bromine, and Chlorine.—Ad. Carnot.—(See p. 67).

On the Mixture of Gases.—A. Leduc.—The author proposes as a new law for the mixture of gases—"The volume occupied by a gaseous mixture is equal to the sum of the volumes which would be occupied by the gases which form it at the pressure and temperature of the mixture." And, further, he affirms that, "in a mixture, each gas should be considered as being subject to the total pressure." In applying this rule to finding the density of argon, he found it to be 19.80, instead of 19.94 as found by Lord Rayleigh and Prof. Ramsay.

Determination of the Density of Gases on very Small Volumes.—Th. Schloesing, jun.—This paper cannot be followed without the accompanying diagram. The author found, however, that his results were of much greater accuracy than he expected.

Spectrum of Cadmium in Vacuo.—Maurice Hamy.—The cadmium lamp, already described, gives at least

twenty lines when the tube is heated to about 295° in vacuo. Some produce interference lines; of these, λ 644 is one. Its wave-length has been determined with great care by M. Michelson's method. Other lines have also been carefully measured, and are here given in a table.

Electrical Resistance of Crystallised Silicon.—Fernand le Roy.—Sticks of agglomerated silicon can easily be prepared of 40 sq. m.m. sectional area and 10 c.m. in length, having a total resistance of from 25 to 200 ohms, according to the variation in pulverisation, compression, and baking. The resistance of silicon, like that of carbon, decreases on heating. At a temperature of 800° the resistance diminishes 40 per cent.

Some New Compounds of the Cerite Metals.—M. André Job.—Will be inserted in full.

Aldehyde of Ammonia.—M. de Forcrand.—The interesting discovery by M. Delépine last month on the constitution of aldehyde of ammonium, has prompted the author to make known some results he obtained ten years ago. Pure and recently prepared aldehyd gave, when dissolved in water, +4.41 cal. at about +12°. Its neutralisation by NH_3 (17 grms. in 2 litres) gave +3.59 cal. after one minute; after that the temperature continues to rise for fifteen minutes, when he again found +3.60 cal., making a total of +7.19 cal. In a further series of experiments, perfectly pure solid aldehyde of ammonia, $\text{C}_2\text{H}_7\text{NO}$, gave as heat of solution +0.1 cal. for one molecule dissolved in 4 litres of water at +12°.

Oxidation of Ammoniacal Compounds by Ferments in the Soil.—E. Demoussy.—The study of nitrification in arable soils leads one to admit that the humus passes to the state of ammonia before being able to become nitrified, and that it is the resistance that it offers to the agents of decomposition that is the cause of the slowness of the formation of nitrates in soils. In some experiments made with methylamine in the presence of ferments in arable soil, nitrites appeared after the sixth day; being replaced by nitrates after a fortnight. The author concludes that, under the influence of the ferments in the soil, the amines are simplified, and by oxidation form ammonia, which only is able to pass directly to the state of nitrous or nitric acid.

Preparation of Gentianose.—E. Bourquelot and L. Nardin.—The authors have prepared gentianose from the fresh roots, in crystalline plates containing no water of crystallisation. It gives colourless aqueous solutions, and burns without leaving any residue; it melts at 207–209°; it is dextro-rotatory to polarised light, $\alpha_D = +31^\circ 25'$ in aqueous solution.

Journal de Pharmacie et de Chimie,
Series 6, vol. vi., No. 12.

Colour of Amorphous Mercuric Iodide.—Maurice François.—Already inserted in full.

Basic Ortho-antimonates of Potassium. — A. Delacroix.—A solution of 1 part of $\text{Sb}_2\text{O}_5 \cdot \text{K}_2\text{O}$ and 2 parts of KHO was evaporated down on a water-bath at 100° in vacuo, and crystals were formed. These were separated from the alkaline mother-liquor and dried in vacuo on a porous plate. The Sb_2O_5 was estimated in the crystalline powder thus obtained by precipitating as sulphide; after moistening with a little HCl the potash was estimated by Rose's method; the water and a little CO_2 were estimated by difference. The results found were:— $\text{K}_2\text{O} = 25.30$ per cent, $\text{Sb}_2\text{O}_5 = 58.44$ per cent, $\text{H}_2\text{O} + \text{CO}_2 = 16.26$ per cent, which gives for the anhydrous salt free from CO_2 the formula $\text{Sb}_2\text{O}_5(\text{K}_2\text{O})_{1.47}$. This leads the author to think that the neutral meta-antimonate does not exist. The ortho-salt, when recently precipitated and prepared in the cold, loses one-third of its potassium when treated with CO_2 , and gives a neutral ortho-antimonate, $\text{Sb}_2\text{O}_5(\text{K}_2\text{O})_{\frac{1}{2}}$.

Impurities in Raw Copper.—M. Schlagdenhauffen.—After leaving some filings of Chili copper in water for several days, then treating this water (after filtration and slightly acidulating it) with sulphuretted hydrogen, a yellow precipitate was obtained. If carried out on a water-bath, a more abundant orange precipitate was formed. If, after completely exhausting with water, the same sample is treated with potash or with weak hydrochloric acid a considerable precipitate of sulphide of arsenic and sulphide of antimony was obtained in the same manner. Other experiments have given all the indications of the presence of selenious acid and lead.

Series 6, vol. vii., No. 1.

Contribution to the Study of the Glycerophosphates.—A. Astruc.—The author confirms previous observations, that the glycerophosphates of lime in solution are alkaline to methyl-orange, and are sometimes acid and sometimes alkaline to phthalein. To a known volume of a solution of glycerophosphate is added enough sulphuric or hydrochloric acid to give a neutral reaction with methyl-orange; then, with a titrated solution of soda, the quantity of alkali is estimated to give neutrality with phthalein. From this it is easy to deduce the quantity of P_2O_5 contained in the solution. The author concludes that the phosphoric acid in glycerophosphates can be estimated very closely and rapidly by the method he describes; that the glycerophosphates of lime appear to decompose, even during their preparation; and that undecomposed glycerophosphate of lime seems to require a quantity of acid equivalent to that of the soda necessary to act on phthalein in order to react on methyl-orange.

MISCELLANEOUS.

City and Guilds of London Institute.—The students of Finsbury Technical College will hold their Annual Conversations at the above College on the 18th inst. Dr. Thompson has promised to lecture on "Wireless Telegraphy"; Mr. Innes will give an exhibition of Colour Photography; and Glow-lamp Making will be demonstrated by Mr. Robertson.

International Congress of Hygiene and Demography.—This Congress will be held in Madrid from the 10th to the 17th of April, 1898, under the patronage of H.M. King Alfonso XIII. and H.M. the Queen Regent, and under the presidency of the Minister of the Interior. The two colonial wars in which Spain has been lately engaged have no doubt interfered to some extent with the preliminary work of the Organisation Committee, but the Secretary trusts that this will not interfere with the complete success of the undertaking. The Congress will be divided into two classes:—Class I., Hygiene, subdivided into ten sections; and Class II., Demography, subdivided into three sections. In all of these sections papers will be read, followed by discussions. There will also be an Exhibition in connection with the Congress, divided into ten classes as follows:—(1) Didactic hygiene; (2) Prophylaxis of transmissible diseases; (3) Urban hygiene; (4) Hygiene in relation to dwelling-houses; (5) Hygiene in what refers to exercise and work; (6) Naval and military hygiene; (7) Hygiene of infancy and schools; (8) Food and dressing; (9) Demography and Statistics; (10) Miscellaneous. Persons willing to be registered as exhibitors should apply to the General Secretary of the Organising Committee, who will immediately refer their requests to the Exhibition Section. Prizes will be awarded consisting of medals and diplomas. The General Secretary is Dr. A. Gimeno, from whom all further information can be obtained.

The Philadelphia Museums.—These Museums had their origin in the successful movement to secure the vast exhibits of natural products from the numerous countries represented at the Chicago Exhibition of 1893. These exhibits were presented to the municipality of Philadelphia, who have devoted a large sum of money to their proper installation. In addition to the Exhibition there is the Scientific and Commercial Library, with its free reading-room, where may be found a large number of statistical and scientific works, valuable to both producers and consumers. A Laboratory has also been started as an adjunct of the Scientific Department of the Philadelphia Commercial Museum, its main object being the examination and analysis of raw materials and manufactured products. The system of this laboratory, which is free to all, will conform closely to that in use in England and on the Continent, and all tests will be made independently in duplicate by two observers, thus ensuring a high standard of accuracy.

MEETINGS FOR THE WEEK.

- MONDAY, 14th.**—Society of Arts, 8. (Cantor Lectures). "The Principles of Design in Form," by Hugh Stannus, F.R.I.B.A.
- TUESDAY, 15th.**—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
- WEDNESDAY, 16th.**—Society of Arts, 8. "The Protection of Industrial Property," by J. F. Iselin, M.A., LL.M. Microscopical, 6. "An Essay in Micro-crystallography" (with Lantern Illustrations), by T. C. White. Mr. J. E. Barnard will give an exhibition of Miscellaneous Lantern Slides.
- THURSDAY, 17th.**—Chemical, 8. "Some Lecture Experiments," by J. Tudor Cundall, B.Sc. "Observations on the Influence of the Silent Discharge on Electricity in Air," by W. A. Shenstone and W. T. Evans. Royal Institution, 3. "Some Italian Pictures at the National Gallery," by Jean Paul Richter. Society of Arts, 4.30. "The Plague in Bombay," by Herbert Mills Birdwood, C.S.I., M.A., LL.D. (This meeting will be held in the East Conference Hall, of the Imperial Institute, South Kensington).
- FRIDAY, 18th.**—Royal Institution, 9. "A Yorkshire Moor," by Prof. L. O. Miall, F.R.S.
- SATURDAY, 19th.**—Royal Institution, 3. "The Structure of Instrumental Music" (with Musical Illustrations), by William H.adow, M.A., B.Mus.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Normantine.—A correspondent wishes to know if there is a chemical combination or preparation called "normantine."

ERRATUM.—The paragraph commencing "It is proper," immediately following Table II., p. 49, should follow Table III., p. 50.

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Superintendent of the Laboratory:

Dr. ALEXANDER SCOTT, M.A., D.Sc.

This Laboratory, which has been founded by Dr. LUDWIG MOND, F.R.S., as a Memorial of Davy and Faraday for the purpose of promoting original research in Pure and Physical Chemistry, is now open.

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All persons desiring to be admitted as workers, must send evidence of scientific training, qualification, and previous experience in original research, along with a statement of the nature of the investigation they propose to undertake.

The terms during which the Laboratory is open are the following—

MICHAELMAS TERM—First Monday in October to Saturday nearest to the 18th of December.

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CONTENTS.

ARTICLES:—	PAGE
The Revival of Alchemy in France, by H. Carrington Bolton, Ph.D.	73
On the Decomposition of Auric Chloride in Dilute Solution, by E. Soestadt	74
Estimation of Mineral Matter in Rubber Goods, by L. de Koningh	74
On some New Compounds of the Cerite Metals, by André Job ..	75
The Principal Amide of Sugar-cane, by Edmund C. Shorley	75
PROCEEDINGS OF SOCIETIES—	
CHEMICAL SOCIETY	76
PHYSICAL SOCIETY	81
ROYAL INSTITUTION	82
EDINBURGH UNIVERSITY CHEMICAL SOCIETY	82
CORRESPONDENCE.—Camphor and its Derivatives	82
CHEMICAL NOTICES FROM FOREIGN SOURCES	82
MISCELLANEOUS	83
MEETINGS FOR THE WEEK	84

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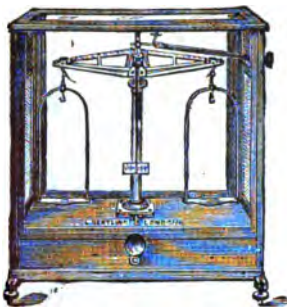
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THE CHEMICAL NEWS.

VOL. LXXVII., No. 1995.

THE REVIVAL OF ALCHEMY IN FRANCE.*

By H. CARRINGTON BOLTON, Ph.D.

(Concluded from p. 70).

For a student of chemistry to read, digest, and write down in intelligible language, in a limited space, the principles of this new school of chemical philosophers is a difficult task, even for one somewhat familiar with the literature of the ancient alchemists; consequently the following analysis falls far short of the ideal. It is properly the work of a kabalist, a theosophist, and a magician, proud designations which the writer disclaims. The modern alchemists accept all the traditions of their ancient predecessors, but give them a new significance, and interweave the novel phenomena derived from researches in pure science. They claim that during the fourteenth, fifteenth, and sixteenth centuries the official schools of instruction taught exclusively the physical part of the sciences, and that the metaphysical part (which is the real life and soul of the study) has been rejected under the opprobrious name of occult science. This living aspect of science has, however, been studied in the secret societies of the initiated, which have preserved the traditions of the kabala, the mysteries of hermetism, and the practice of transmutation. The study of science is as much a religious question as an intellectual one, and worship at an altar should sustain and enlighten the worker in a laboratory. "Chemistry, alchemy, and hermetic philosophy form three steps of the ladder which leads the initiated from the laboratory, through artistic realisation, to the oratory: '*Labora, Opera, Ora et Invenias.*'"

The modern alchemists also maintain that Darwin and his disciples appreciated but a small part of the great doctrine of evolution, which should be applied to the chemical elements as well as to living beings. The starting point in the evolution of elements is the ether (the universal astral fluid of the kabalist), the infinitely divisible particles of which form chemical atoms by agglomeration. This ether is condensed energy, and hence all matter is resolved into energy.

Energy, matter, and motion form a trinity analogous to the Divine Trinity, one in substance, three in appearance. Matter is one in kind, and the diversity of chemical bodies results from differences in grouping and in motions of the constituent particles. Intelligence is allied in a mysterious way with matter and energy, forming another trinity. Every atom centralises intelligence, is in itself a living entity, and by a process of self-evolution yields the diverse natural bodies. "Ether is the father of hydrogen, from which are derived oxygen, nitrogen, carbon, &c., combinations due to etheric vortices." "Perhaps helium should precede hydrogen." This view of matter as a living entity is greatly insisted on, and the doctrine is called *Hylolism*. An alchemist who expects to succeed must possess psychic power over atoms, so that by the action of his will they shall group themselves to form the metal desired.

Such is the physical philosophy of modern alchemists; the kabalistic philosophy is by no means so clear, being closely linked to the *Tarot*, which signifies the "hieroglyphs and algebraic calculations of the Primordial Genesis."

* Abstract of a Paper read to New York Section of the American Chemical Society, October 1, 1897. From *Science*, N.S., vol. vi., No. 134.

Students of the mystical philosophy of the Hebrews discover profound occult significance in accidental similarities of widely differing objects and phenomena. The seven planets, seven days of the week, seven colours, seven orifices in the head, seven metals known to the ancients, seven archangels, and seven infernal demons present to the truly kabalistic mind marvellous and precious analogies. In the "Table of Concordance of Major Arcana" these correspondences are given: "Heth—Justice—Elementary existence—Nizah—Cancer—June—Hydrogen—Fire."

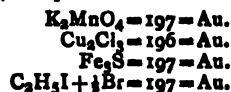
Jollivet-Castelot has written of Kabalistic Alchemy, and a perusal of his essays leaves in the mind of the uninitiated confused memories of colours, numbers, signs of the zodiac, alchemical operations as distillation, fixation, and the like, the names of the sons of Jacob, certain precious stones, geometrical figures, Hebrew characters, Azoth, quintessence, and the Devil, all discussed in a language as obscure as the symbolism portrayed. To conceal esoteric mysteries abbreviations are often used, but one does not have to be very deeply initiated to recognise in P. P. Ph. "la pierre philosophale," and in G. O. "grand oeuvre."

Astral light is an important factor in modern hermetism, and is related very closely to the "radiant matter" of chemists and the "ether" of physicists. "Astral light is the universal agent, the universal plastic mediator, the common receptacle of vibrations of motion, and of the phantoms of form."

Writers on modern alchemy discuss the marvels of palingenesis, of homunculi, and of gamahes; they write of the materialisation of a metal through the medium (mediumité) of a metal; they cite the "Rules of Philalethes," the works of George Ripley and of the Cosmopolite, and refer in the same essay to Berzelius, Berthelot, and Moissan. We are told that "*le diable est le singe de Dieu*," and that the "Cherubim of the Ark of the Covenant symbolises the male and the female of the Universe, the Alchemical Father and Mother," by the very authors who show acquaintance with the most recent advances in pure chemistry. In liquid fluorine they perceive a realisation of the Alkahest, or universal solvent long sought by mediæval alchemists.

Accustomed to juggle with numbers, the kabalist finds abundant opportunity in the atomic weights of the elements, and he makes the most of his opportunity. When the arithmetical sum of the atomic weights of elements entering into a given compound chances to equal the atomic weight of gold, this accidental correspondence is seized upon as a pretext for claiming hermetic relationship between the two substances.

August Strindberg has devoted much study to such correspondence, and points out the following:—



He uses both 196 and 197 as the atomic weight of gold to suit his purposes, and seems to be very weak in arithmetic, for the hypothetical body Fe_2S has a molecular weight of 200.

The ammonium-ferrous-sulphate crystallised with six molecules of water, which chances to have a molecular weight equal to that of gold, is used by Strindberg as basis of the following experiment, which serves to show his method of reasoning and of operating.

Ammoniacal sulphate of iron = 392 = Au is perhaps the solution of the enigma; sulphate of iron (green copperas) precipitates solutions of gold; to precipitate according to monist-chemistry is to enter as a factor into the re-constitution of the body of a compound. Soak a strip of paper in a solution of sulphate of iron, and expose to the fumes of ammonia; the paper will assume a bluish-green colour like that of the protoxide of gold. Dry the strip of paper over a lighted cigar, and the paper will acquire a chestnut-

brown colour like that of the deutoxide of gold. Little by little metallic flakes of a golden yellow colour appear, forming a non-solid (*non-fixé*) gold, when the sulphate of iron produces an auto-lecondation by self-precipitation. However, the golden flakes amalgamate with mercury, which property is not shared by iron. After showing by appropriate tests that iron is still present, the hermetic chemist proceeds to explain the reaction by assuming the formation of the hypothetical $\text{Fe}_2\text{S}=196=\text{Au}$, or of the imaginary compound,



or of the



and he adds, "The chloride of gold is reduced by the nicotine of the cigar." Since, however, no reagent containing chlorine in any form was used in the experiment, this element must have been created at the same time with the gold, which, however, is "incomplete" gold soluble in unmixed acids.

To acquire knowledge and power for successful hermetic labours, to become eligible for initiation in the occult societies, is no easy task. The aspirant must strive valiantly against the passions that assail him, casting out of his soul pride, anger, jealousy, hatred, avarice, hypocrisy, idleness. If the candidate for honours desires to become worthy of the name hermetic philosopher he must prove himself a Magian; he must learn to exercise his will on matter in all its forms, and to acquire this power he should practice crystal-gazing and reading in magic mirrors; to learn to perceive the invisible he must withdraw from the visible, imposing on himself psychic sleep, called by some hypnosis. As adjuncts to the attainment of the ideal mental state he should use perfumes, music, and light; and eventually the astral body, separated from the physical body, will supply the intellectual, moral, and material illumination indispensable to the Great Work.

It is rather discouraging to learn that, even after fulfilling all these hard conditions, no one can realise the perfection desired until he has passed through several of our planetary existences. The would-be alchemist must also follow the precepts of Albertus Magnus; he should be discreet, silent, and must not reveal the result of his labours; he must reside in an isolated place, and choose the time and the hours for his tasks; he must be patient, assiduous, and persevering, and he must be rich enough to bear the expenses of his pursuit. Besides ordinary chemical apparatus he should provide several objects indispensable to his work, a magic wand, a sword to dissolve the astral coagulations, a magic mirror, a brazier for perfumes, a wooden altar covered with a white cloth, and an alchemist's robe of white linen to be worn with a girdle embroidered in gold and silver. In all his chemical operations he must project psychic force into the reagents.

Bright prospects for the future of chemical science are claimed by this school of philosophers. Inorganic chemistry is destined to follow the lines in which inorganic chemistry has prospered; the formation, derivation, or rather the evolution of metalloids (so-called) and of metals will be realised through etheric cyclones, different degrees of condensation of hydrogen. Chemical bodies are of one kind only, and they are all organic and living.

There is a growing belief among advanced chemists in the theory that the elementary bodies as known to us are compounds of a unique primary matter (*protyle*), and that transformation of one kind into a similar one is not beyond the bounds of possibility, but we do not think that the modern hermetists are pursuing the right path to accomplish this end; nor do we believe that the world of science is any nearer the coveted goal of alchemical avarice.

In conclusion, a passage from Lord Bacon: "I was ever of opinion that the philosopher's stone and an holy

war were but the rendezvous of cracked brains that wore their feather in their heads."

ON THE DECOMPOSITION OF AURIC CHLORIDE IN DILUTE SOLUTION.

By E. SONSTADT.

It is stated in chemical text-books that solution of auric chloride is decomposed by exposure to light with separation of gold. This reaction I have not obtained, possibly either because my solution was not exposed to light long enough, or because it contained free hydrochloric acid—as ordinary auric chloride, dried on the water-bath, was used. But the same solution which failed to react with moderate exposure to light gave a precipitate of gold on heating for some hours. I then dissolved 1 part of the chloride in about 15,000 parts of water which had been first distilled in the usual way, and was then coloured by addition of a small proportion of bichrome, and again distilled in a glass retort adapted by grinding to a glass receiver, every care being taken to exclude organic matter. The solution behaved on heating exactly as in the former case, gold being precipitated. In both cases a trace of hydrogen peroxide was detected in the solution after the reaction had taken place. If the reaction took place without heating, it is reasonable to suppose that the H_2O_2 produced would be much more easily recognisable.

The reaction appears to be entirely analogous to that which I have shown to take place when platinum chloride is similarly heated in dilute solution (see p. 79; also *Proc. Chem. Soc.*, 1898, p. 25). In the latter case the reaction is expressed by the equation—



while the corresponding equation for the auric chloride would be—



But as aurous chloride decomposes, as is well known, when heated with water, into gold and auric chloride, gold only is precipitated.

It seems probable that the indicated reaction is a general one for the higher chlorides of metals of the platinum group.

ESTIMATION OF MINERAL MATTER IN RUBBER GOODS.

By L. DE KONINGH.

It is a well-known fact that the amount of mineral matter in rubber goods cannot be determined by means of a simple ignition, as the ash does not represent the original mineral matter. For instance, white-lead becomes lead oxide, chalk is largely reduced to calcium oxide, some oxides volatilise, &c. By using the following process the writer believes he has effected a decided improvement:—Five grms. of the very finely divided sample are treated in a covered beaker with 50 c.c. of fuming hydrochloric acid, and after soaking for an hour the whole is heated up to 70° for another hour; 50 c.c. of water are then added, and the insoluble matter is collected on a filter. The washing with boiling water takes some time, as the acid causes the rubber to swell out to an enormous extent; however, after a litre of water has been used the washings are generally free from acidity. The residue is now carefully transferred from the filter, into a weighed porcelain dish; no practical difficulty will be experienced in this operation. After drying on the open water-bath until no more water-vapour is visible, the dish is placed inside an

air-bath and heated for three hours up to 105°. After cooling it is weighed, and the result is the rubber minus the greater part of its soluble ash. The remainder of the ash is then found by an ignition. Some samples examined by the writer, which gave on simple ignition about 50 per cent of ash, gave only 10 per cent after the acid treatment, and the ash was then found to consist chiefly of barium sulphate. If it be desired to distinguish between heavy spar and silicates, the writer still uses a process described by him some years ago, based on the fact that barium sulphate is soluble in hot sulphuric acid, and may be completely re-precipitated on adding water.

The acid filtrate may, if desired, be subjected to further analysis.

Before recommending the process it was necessary to ascertain if the strong acid dissolves any organic matter. If the filtrate is evaporated nearly to dryness after having added a few c.c. of sulphuric acid, there is obvious darkening, and an unpleasant albuminous odour is noticed. But, as will be seen, the loss is not very great, and may even to some extent be allowed for. Five grms. of a block-rubber containing 40 per cent of ash were treated with 50 c.c. of fuming hydrochloric acid at 70°; 50 c.c. of water were then added, and after standing for several hours 50 c.c. of the filtrate were diluted with 450 c.c. of water, and 30 grms. of crystals of magnesium sulphate were added on account of the presence of hydrochloric acid. Standard solution of potassium permanganate (0.001 gm. oxygen per c.c.) was added until the liquid turned pink, and more was added during the next half-hour; each time the colour began to fade. Three c.c. of permanganate were thus consumed. To ascertain how much organic matter this represents 0.03 gm. of a dry albumenoid (peptone) was dissolved in 25 c.c. of fuming hydrochloric acid, and then titrated with permanganate in exactly the same way; the result was also 3 c.c.

By an easy calculation it follows that 5 grms. of the block-rubber had yielded to the acid about 0.06 gm. of organic matter, or about 1 per cent.—*Journal of the American Chemical Society*, xix., No. 12.

ON SOME NEW COMPOUNDS OF THE CERITE METALS.

By ANDRÉ JOB.

THE oxalates of the cerite metals are soluble in warm concentrated hydrochloric acid. If we let the solution cool slowly to the ordinary temperature, we notice that fairly large, well-defined crystals are deposited. They differ completely in their composition from the original oxalate. In fact, if we calcine them at a red heat and dissolve the residue from the calcination in nitric acid we can perceive the odour of chlorine.

Having made these observations, I was desirous of studying more closely the compound which was formed, and I took the salt of lanthanum as my first subject for analysis. According to my experience the constant composition of the crystals of the oxalochloride is shown by the following formula:— $(\text{C}_2\text{O}_4)_2\text{Cl}_2\text{La}_2 + 5\text{H}_2\text{O}$.

We can see that it is an oxalate where 1 molecule of oxalic acid has been displaced by 2 molecules of hydrochloric acid. Boiling water decomposes this oxalochloride into insoluble oxalate and soluble chloride:—



The crystals obtained as above were kept for several hours at 120° without undergoing any decomposition. At 230° they lose their water of crystallisation. We note the loss of weight. We dissolve in cold nitric acid and precipitate the solution with nitrate of silver, and thus estimate the chlorine. I assured myself that concentrated nitric acid drives off all the chlorine on boiling, and that on evaporating and calcining we obtain a residue of

oxide; in this manner I estimated the lanthanum. Finally, I estimated the oxalic acid in the cold in nitric solution, and, in spite of the presence of hydrochloric acid, by means of an oxidising solution of cerium. I shall shortly describe this new method of analysis.

The analysis of this compound does not require the lanthanum to be absolutely free from cerium and didymium. Still I purified the sulphate used as a starting-point with great care. The spectroscope, even, did not show the presence of didymium. I also proved the absence of cerium, for which I have found a new and very sensitive reagent; the salts of the cerite metals are nearly all very soluble in a concentrated solution of neutral carbonate of potassium. One drop of peroxide of hydrogen shows the presence of cerium by an intense red colouration if it is present in any notable quantity, and by as distinct a yellow colour even if the solution is diluted down to 1 part in 250,000.

After having analysed the oxalochloride of lanthanum, I prepared the analogous compounds of cerium and didymium, and made the analyses. I also decided that oxalobromides and oxaliodides exist. The oxalochlorides of the cerite metals possess one very interesting property; the oxalochloride of lanthanum calcined at a red heat does not lose its chlorine, and the composition of the residue is similar to that of the original oxalochloride, $\text{La}_2\text{O}_2\text{Cl}_2$. Thus we have a convenient method for the easy preparation of the oxalochlorides of these metals.

There is an opportunity to study, from the point of view of chemical equilibrium, the formation of the oxalochlorides in dilute hydrochloric solution. I have observed that the oxalates precipitated from a hydrochloric solution, even when dilute, still retain a considerable quantity of oxalochloride; and thus it may be explained that different chemists, in calcining the oxalates and the sulphates, have constantly found different atomic weights. It remains to be seen if, in starting with oxalochloride itself, we cannot manage to obtain concordant results. I would here recall the fact that Clève prepared an oxalonitrate of didymium, and that Wyruboff and Verneuil have notified the presence of oxalonitrite of cerium in the oxalate crystallised from a concentrated nitric solution.

I would finally add that Souchay and Leusenn have prepared an oxalochloride of calcium. Calcium presents, in the same manner as the cerite metals, this common property, that its oxalate is insoluble in dilute acids. I am following up the research on the metals whose oxalates are soluble; this will be the subject of another communication.—*Comptes Rendus*, vol. cxvii., No. 3.

THE PRINCIPAL AMIDE OF SUGAR-CANE.

By EDMUND C. SHORREY.

It has been more than forty years since Lawes and Gilbert pointed out that in plants used as feed for stock, part of the nitrogen exists in the amide form. Since then several schemes of analysis have been devised by which to determine the amounts of different forms of nitrogen in plants, and numbers of analyses have been published in accordance with these schemes, so that it has come to be generally accepted by chemists that a part of the nitrogen of all plants, in the growing stage at least, is in the amide form. E. Schulze, who has done more than anyone else to advance our knowledge of the forms in which nitrogen exists in plants, states that the amide compounds vary with different plants, and also with the age and condition of the plant.

In 1892 I determined the total and albumenoid nitrogen content of a number of samples of mature sugar-cane. In the samples then examined I found the albumenoid to be about 90 per cent of the total nitrogen. Analyses made later, of another and less mature variety of cane,

showed this non-albumenoid nitrogen to be sometimes as high as 25 per cent of the total nitrogen. The albumenoid nitrogen was determined by precipitating with cupric hydroxide, taking special precautions to prevent any decomposition of the albumenoids by preliminary heating. No attempt was made at that time to determine the character of the non-albumenoid nitrogen.

In January, 1894, a paper was read by W. Maxwell before the Louisiana Sugar Planters' Association on "Organic Solids not Sugar in Cane Juice." The matter of this paper was subsequently issued as a bulletin by the Louisiana Experiment Station. In this paper attention was drawn to the fact already noticed, that all plants contain at some period part of their nitrogen in the amide form—a fact well known to chemists, but apparently overlooked by sugar manufacturers. After giving a number of analyses of cane-juice, in which the difference between the total and albumenoid nitrogen was designated amides, Maxwell stated that the amide of cane-juice was found to be asparagin, crystallised preparations of this body having been obtained. He made, however, no statements regarding the physical or chemical properties of the asparagin obtained, nor did he give any information as to how it was ascertained to be asparagin.

Some months after the publication of this paper, there was a discussion in the *Bulletin de l'Association des Chimistes de Sucriers*, between H. Pellet and W. Maxwell, as to the possibility of the asparagin of cane-juice affecting the polariscopic reading of such solutions. In view of this possibility I determined to make some preparations of the amide compound or compounds in cane-juice, and ascertain the rotatory power of the same. This work was begun in January and continued until July of last year, and covers every variation in age and condition of cane delivered at the mill last year, as well as some samples of very young cane.

As a result of this work I have found that the principal amide compound present in sugar-cane is *not optically active*, and is *not asparagin*, but glyccoll or glyccin, an amide not heretofore known to occur in plants.

The method of separating the amide was that commonly used, viz., precipitation with mercuric nitrate. To the juice obtained from the cane sample by pressure, a slight excess of lead subacetate was added, the liquid filtered, and to the filtrate an acid solution of mercuric nitrate was added, and the whole brought to faint acidity if strongly acid, by the cautious addition of caustic soda. The precipitate thrown down, which was white and flocculent, after being well washed, was decomposed by hydrogen sulphide. The filtrate from the mercuric sulphide, after being concentrated to a thin syrup, was allowed to stand, when abundant well-defined crystals were generally formed in thirty-six hours. The filtrate from the mercuric sulphide was always quite strongly acid, and the subsequent crystallisation was found to be much more rapid and the yield larger if the solution were neutralised with ammonia before concentration, and neutrality maintained during concentration by the continued addition of ammonia as the solution became acid again. This addition of ammonia made no difference in the character of the crystalline body obtained. The crystals after separation from the mother-liquor were purified by re-crystallising twice.

The mercuric nitrate used was prepared as wanted by boiling mercury with an excess of nitric acid until no further reaction for mercurous nitrate was obtained. I have as yet made no analysis of the white insoluble precipitate which mercuric nitrate gives with the amide body in question.

The purified crystals were in the form of plates or four-sided prisms, belonging to the monoclinic system; glassy in appearance, quite hard, grating between the teeth, and having a sweetish taste. They were soluble in cold water, but much more readily in hot, soluble in 80 per cent alcohol, but insoluble in ether. In all, fifteen preparations of these crystals have been obtained, and in all

cases the physical properties of the crystals and the chemical properties of the solution have been the same.

The mother-liquor from the crystals was found to contain other nitrogenous bodies not readily obtained in a crystalline form, the nature of which has not yet been ascertained; but they are comparatively small in amount, and, since nearly the whole of the bodies precipitated from cane-juice by mercuric nitrate can be obtained in a single crystalline form having the properties of an amide, it is quite justifiable to speak of this body as the principal amide of sugar-cane.

In examining this sugar-cane amide to ascertain if it were optically active, solutions containing from 2 to 4 grms. of the substance per 100 c.c. of water were examined in a 200 m.m. tube of a Schmidt and Haensch half-shade polariscope, observations being also made on similar solutions after the addition of caustic soda or nitric acid. In no case was any rotatory power shown. As asparagin is slightly left-handed in water solution, more strongly so in alkaline solution, and strongly right-handed in acid solution, it was at once evident that the sugar-cane amide must be an inactive form of asparagin, or some other body not asparagin. A more extended examination soon brought out other points of difference between the sugar-cane amide and asparagin; and on comparison of the properties of this amide with those of the other known bodies, it was shown unmistakably to be glyccoll.

The chief points of difference between asparagin and the sugar-cane amide may be stated as below:—

	Sugar-cane amide.	Asparagin.
Optical activity.	Inactive.	Left-handed in water solution.
Water of crystallisation.	None.	One molecule lost at 100° C.
Behaviour with Fehling's solution.	Does not reduce on boiling.	Reduces on boiling.
Behaviour when boiled in alkaline solution.	Gives off NH ₃ only if alkali is quite concentrated, leaving HCN in solution.	Gives off NH ₃ , leaving aspartic acid in solution.

It will be noted that these properties of the sugar-cane amide are identical with those of glyccoll, and, in addition to this correspondence, both produced a grey precipitate of metallic mercury when added to a solution of mercurous nitrate, and both give a red colouration with ferric chloride.

The reaction by which the sugar-cane amide is most readily distinguished from asparagin, and by which also its identity with glyccoll is thoroughly established, is that which results on heating in alkaline solution. As is well known, asparagin gives off ammonia when heated in quite dilute alkaline solution, leaving aspartic acid in solution. On the other hand, glyccoll and the sugar-cane amide do not give off ammonia when heated in alkaline solution unless such solution be strongly alkaline, and after the evolution of ammonia has ceased no aspartic acid is found in the solution, but if hydrochloric acid be added to acidify and the solution heated, hydrocyanic acid is given off and can be detected by the smell, and oxalic acid is left in solution and can be precipitated as calcium oxalate. When operating with very small quantities of the amide, as I often found it necessary to do, the quantity of hydrocyanic acid given off in this reaction is so small that the smell is masked by that of the hydrochloric acid; in this case the presence of hydrocyanic acid can be established in the usual way by placing a drop of yellow ammonium sulphide on a porcelain dish, holding over the boiling solution for a few seconds, removing the excess of sulphide by blowing, and proving

the presence of a thiocyanate by the red colouration produced on addition of ferric chloride.

The sugar-cane amide also gives off hydrocyanic acid when heated with dilute sulphuric acid and manganese dioxide.

When heated in a sealed tube with benzoic acid, the sugar-cane amide gives hippuric acid, a condensation characteristic of glycocoll, and one by which it is supposed hippuric acid is formed in the animal body.

The hippuric acid so formed was identified by separating it from any remaining benzoic acid by agitation with petroleum ether, evaporating the insoluble residue to dryness with nitric acid and heating, when the characteristic smell of nitrobenzol was detected.

Glycocoll has been prepared in the usual way from hippuric acid by boiling with dilute sulphuric acid, and the reactions given above as characteristic of that body have been verified throughout.

It having been thus thoroughly established that the principal amide of sugar-cane is glycocoll, it is well to note that there are certain points in which glycocoll resembles asparagin, and the resemblance is such that anyone prone to jump at conclusions would, on obtaining a crystallised preparation of the sugar-cane amide, immediately pronounce it asparagin. The points of resemblance are these:—First, the general appearance, solubility, &c., of the crystals; second, neither gives up any nitrogen when treated with sodium hypobromite in alkaline solution; third, both dissolve cupric hydroxide to a blue solution; and fourth, both contain the same per cent of nitrogen. Asparagin crystallised with 1 molecule of water of crystallisation and glycocoll each contain theoretically 18.66 per cent nitrogen, while the average of all samples of the sugar-cane amide was 18.69 per cent. The Gunning method was used in determining the nitrogen, and clean, well-defined crystals were selected for such determinations.

In all, fifteen preparations of the glycocoll have been made from sugar-cane; and the samples of cane from which it has been prepared have included young shoots of cane a few weeks old, the green tops of cane one year old, and mature cane growing at elevations from 400 to 1200 feet above sea-level. It is fair, then, to conclude that glycocoll is not only the principal amide of sugar-cane, but is also a normal constituent of this plant at all periods of its growth.

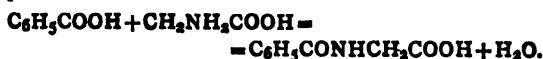
I have as yet made no attempts to estimate the quantity of glycocoll in sugar-cane or any of its products, but have noticed that larger amounts have been obtained from young than from mature cane.

The average nitrogen content from mature sugar-cane is probably about 0.30 per cent, and of this at least 0.22 per cent is albumenoid nitrogen; so that the amido content cannot be higher than 0.08 per cent, and probably is often less than 0.02 per cent. It is easily seen, then, that the preparation of any quantity of the sugar-cane amide entails considerable labour, and this, coupled with the fact that this work has been done at odd times during a busy grinding season, is my excuse for the incompleteness of this research in some respects.

The identification of glycocoll, glycocine, or amido-acetic acid in sugar-cane is of interest from several points of view. Its occurrence has not been noted in a plant before, and it has been considered a body belonging wholly to animal chemistry. It can be prepared from gelatin by heating with acids or alkalis,—in fact was first prepared in this way,—and it has been supposed that it was formed in the animal body from gelatin or gelatin-yielding proteids.

It does not seem to occur free in the animal body, and the theory of its formation from proteids seems to have been demanded by the following fact:—Benzoic acid, when taken into the stomach, appears in the urine as hippuric acid, and to explain this it has been supposed that the condensation already referred to takes place, viz.,

benzoic acid and amido-acetic acid combine to form hippuric acid with the elimination of water:—



This view is strengthened by the fact that when glycocoll is taken with benzoic acid the yield of hippuric acid is increased.

In the urine of herbivorous animals hippuric acid occurs in relatively large quantities, and while the food of such animals has been deemed capable of supplying the benzoic acid part, the source of the glycocoll to form such large quantities of hippuric acid has been more or less of a puzzle to physiologists.

The identification of glycocoll in sugar-cane, and the fact that it has been mistaken for asparagin, suggests the probability of its occurrence in other plants, especially the *Graminae*, which form the major part of the food of herbivorous animals; and it is quite likely that the source of the hippuric acid in the urine of such animals will be found in such occurrence.

In vegetable, as in animal physiology, the question of the constitution of proteids is uppermost. Now glycocoll in the physiology of sugar-cane no doubt plays the same part which other amides are known to do in other plants. It is the form in which nitrogen is conveyed to growing parts, and when maturity is reached the amide becomes the albumenoid to become the amide again when a new growth takes place. A number of facts collected during this study of the sugar-cane amide point to the existence in cane of a gelatin or gelatin-yielding proteid which yields glycocoll as one of the products of decomposition. This, coupled with the well-known and comparatively simple constitution of glycocoll, seems a step toward the understanding of at least one proteid of sugar-cane. The further study of this matter promises to be of special interest, both from a physiological and a technical point of view.

From the sugar manufacturer's point of view the presence of glycocoll presents the following points of interest:—It is known that at various stages of sugar manufacture ammonia is given off from boiling cane-juice, especially if this juice has been rendered alkaline by an excess of lime. This has been stated as due to the decomposition of asparagin into aspartic acid and ammonia. But since we know that the amide is glycocoll and not asparagin, and that glycocoll is not decomposed unless boiled in strongly alkaline solution, it is plain that the ammonia must be derived from the decomposition of albumenoids. When, as is generally the case, cane-juice is maintained approximately neutral throughout the course of manufacture, the whole of the glycocoll originally present in the juice should be found in the final molasses.

Whether this theory always carries out in practice I have not yet ascertained, but I have obtained glycocoll in comparatively large quantities from several samples of refuse molasses by the same method by which it was obtained from cane juice.

In one sample I determined the amounts of different forms of nitrogen according to a well-known scheme of analysis with the following results:—

	Per cent.
Nitrogen as free ammonia	0.011
" albumenoids	0.126
" peptones	0.050
" amides	0.201
" other forms	0.228
Total nitrogen	0.616

Free ammonia was determined by distilling with magnesia free from carbonate, and albumenoids by precipitating with cupric hydroxide. The nitrogen designated peptones is that precipitated by phosphotungstic acid after the removal of free ammonia and albumenoids. In the case of cane juice and its products this precipitate

does not contain peptone bodies, and its character will be treated in a future paper. After removal of these three forms of nitrogen, amides were determined by boiling for an hour with 2 per cent sulphuric acid, neutralising, and distilling with magnesia free from carbonate and doubling the amount of ammoniacal nitrogen obtained for amide nitrogen. This method, which is fairly accurate in the case of asparagin (which gives up half its nitrogen as ammonia when boiled with dilute acids), is of no value in the case of glycocoll, so that the above analysis should read—Amides and other forms, 0.429 per cent, instead of

	Per cent,
Amides	0.201
Other forms	0.228

From a number of years' experience in working with cane juice, I am convinced that the formation of molasses is due to mechanical, rather than chemical, conditions, that the presence of viscous non-crystallisable bodies presents the further crystallisation of sugar by rendering the motion of the sugar molecules in the liquid difficult, and the mellassigenic action of crystallisable salts is very slight. A body such as glycocoll would then exert little effect on the crystallisation of sugar one way or the other, especially as it exists in cane in such small amounts, but it is likely that the proteid directly connected with glycocoll will be found to be highly mellassigenic.

We have been in the habit of associating the so-called gums or viscous bodies in cane juice with the cellulose or non-nitrogenous constituents of the plant, but we may have to modify this view in the presence of a gelatin-yielding proteid peculiar to sugar-cane and allied plants.—*Journal of the American Chemical Society*, xix., No. 11.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 3rd, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

CERTIFICATES were read for the first time in favour of Messrs. Charles Baskerville, North Carolina University, Chapel Hill, North Carolina; Alfred Campion, 637, Alexandra Parade, Dennistown, Glasgow; Robert Martin Caven, University College, Nottingham; F. Hudson-Cox, 67, Surrey Street, Sheffield; John Arnold Fleming, Britannia Pottery, Glasgow; Harry Pearson Hodgson, Caldew Bank, Cummersdale, Carlisle; Edwin Charles Jee, 45, Popsy Road, New Cross, S.E.; Edward Jones, Vine Cottage, Tudor Road, Kingston-on-Thames; Walter Ratcliffe, 21, Mawdsley Street, Bolton; Henry Somerville, 33, Vincent Square, S.W.; Harry St. John, Thornfield, Sunderland.

Of the following papers those marked * were read:—

*8. "The Volumetric Estimation of Sodium." By H. J. H. FENTON, M.A.

Dihydroxytartaric acid, in presence of dilute sulphuric acid, is readily oxidised by potassium permanganate at the ordinary temperature, the reaction affording a very convenient method for the estimation of the acid or its salts. A method has been devised for the quantitative estimation of sodium based upon this relation and upon the sparing solubility of sodium dihydroxytartrate. The solubility of the sodium salt at 0° is shown to be extremely small, and in presence of excess of a dihydroxytartrate is practically negligible.

The substance to be examined, in concentrated neutral solution, is mixed with excess of potassium dihydroxytartrate, and the mixture is kept at 0° for half an hour. The precipitated sodium salt is washed with a little ice-

cold water, dissolved in excess of dilute sulphuric acid and titrated with potassium permanganate.

The results obtained with sodium sulphate, chloride, nitrate, acetate, and with rochelle salt, are accurate within 0.3 per cent. The presence of magnesium does not interfere with the accuracy of the results; but ammonium salts, if present in excess, lead to low results. Full details for working the process are given in the paper.

The potassium and ammonium salts of dihydroxytartaric acid have been prepared and examined, and, together with other derivatives of the acid, will be described in a future communication.

DISCUSSION.

Mr. HEHNER said that analysts would welcome the method. It was remarkable that sodium, the most widely distributed element, had hitherto been without a direct method of quantitative estimation. He would suggest that the process should be conducted gravimetrically.

Mr. JOHN A. R. NEWLANDS said that in some experiments for manufacturing purposes, made a quarter of a century ago, he had used a method in which sodium was precipitated as acid oxalate, using suitable precautions, and afterwards titrated, either before or after incineration. The results, however, were only of an approximate character, and did not approach in accuracy to those obtained by the author of the present paper.

Dr. LAWSON asked how Mr. Fenton's method for the production of dioxytartaric acid compared, as regards yield, with that usually employed. The sodium salt was of technical importance in the preparation of a valuable dye-stuff, and it would be of interest to know whether the results were comparable with those obtained by decomposing nitrotartaric acid.

Mr. FENTON, in reply, said that he had suggested in the paper that the process might be conducted gravimetrically, but he considered that the volumetric method would find most favour. He had experimented with other sparingly soluble sodium salts, but did not think that any could nearly compare with the dihydroxytartrate as regards insolubility. In reply to Dr. Lawson, he said that the process possessed many advantages as compared with the usual method of preparing the sodium salt from nitrotartaric acid.

*9. "The Atomic Weight of Boron." By F. P. ARMITAGE.

The determination of the water of crystallisation was the method used. The values for the ratio $\text{Na}_2\text{B}_4\text{O}_7 \cdot \text{H}_2\text{O}$ obtained by previous workers are not sufficiently concordant to allow of the values for the atomic weight of boron, calculated from them, being regarded as very trustworthy. The discrepancies are probably to be traced to the slight efflorescence which occurs during the preliminary drying of the crystals of borax. A new method of drying was consequently applied, the borax being washed with alcohol and ether successively, and the ether expelled by exposure for six hours in a vacuum. A given weight of the dry crystals was dehydrated in a stream of air, the final traces of water being expelled by fusion over the Bunsen burner and not over the blowpipe.

In a series of six experiments, the greatest difference in the percentage value for the water of crystallisation was 0.413, and the mean atomic weight of the element as calculated from these values 10.959, a number differing by 0.006 from that recently obtained by Ramsay and Aston in their experiments on the distillation of sodium diborate with hydrochloric acid and methyl alcohol.

Certain results are also given in which the method of titration (*cf.* Rimbach, *Ber.*, 1893, xxvii., 164) is applied with a view of ascertaining the possible degree of its accuracy. Thus, a given weight of fused borax was dissolved in water and titrated with dilute sulphuric acid, the strength of which had been determined (i.) by titration with solution of pure soda of known strength, (ii.) as barium sulphate. The value 10.928 was obtained for the

atomic weight of boron as a mean of two experiments performed by this method.

DISCUSSION.

Mr. VELEY said that methods of determination of atomic weights based upon the elimination of water from a crystalline salt presented the difficulties, firstly, of the mechanical inclusion of water within the walls of the crystals, however fine; and secondly, of the possible efflorescence and deliquescence of such salts. These difficulties, as also that of the intumescence of the anhydrous salt, when heated with the blowpipe, induced Ramsay and Aston to reject this method. It appeared that the author had overcome these difficulties to a great extent by removing the adherent water by washing with absolute alcohol and subsequently with ether, which was pumped off, as also by heating the anhydrous salt only at a temperature obtained by the Bunsen burner, and not at that of the blowpipe, when loss occurred either on account of the volatilisation or the decomposition of the borax. The experiments of the author also showed that the method of determining the atomic weight proposed by Rimbach, though not of the degree of exactitude necessary for such determinations, was probably more accurate for the purpose of acidimetry than that of sodium carbonate, which was not readily obtained of the composition required by its molecular formula.

Mr. GROVES asked whether the volatilisation observed when the crucible was heated in the blowpipe flame might not be due to the action of water vapour and carbonic anhydride which would diffuse through the perforated cover.

Dr. SCOTT was astonished that anyone now should attempt to determine atomic weights through calculations of the amount of water of crystallisation contained in hydrated salts. The experiments of J. W. Mallet on ammonia alum, and of many other experimenters, had shown how unsatisfactory such determinations must always be. It was evident that as such salts produced a determinate vapour pressure of water depending on the temperature, if the air in which they were dried contained less than that amount the salt must lose water during its drying.

The PRESIDENT considered it superfluous to record weighings to six places and the atomic weight to three places of decimals, having regard to the method adopted. With reference to the process employed by the author in drying the borax, he failed to understand why, if borax is an efflorescent salt, it loses no water of hydration when washed with absolute alcohol. He suggested that in investigating this point the influence of time should be taken into account.

Mr. ARMITAGE, in reply, said that he had not examined the sublimate formed on the lid of the crucible when ignited with the blowpipe, as his object was to show merely that a loss of weight would occur if this method is adopted. The standard of atomic weight taken was oxygen = 16. The time occupied for each washing by absolute alcohol was three to four minutes.

*10. "Rate of Escape of Ammonia from Aqueous Solution." By E. P. PERMAN, D.Sc.

If a current of air is drawn through a dilute aqueous solution of ammonia at a uniform rate, the amount of ammonia q in solution when a volume V of air has passed through the solution, is accurately represented by the equation $\log q = a - bV$, a and b being constants. The volume of air was measured by a meter.

The equation does not express the results given by a concentrated solution; the solution used in most of the experiments contained 70 grms. of ammonia per litre.

A number of experiments, conducted at different temperatures, prove that the logarithmic formula holds good between 0° and 46°; it was also found to hold good when the pressure was varied.

The value of b is a measure of the rate of escape of the gas from the solution under any specified conditions;

such values were obtained at various temperatures, and a simple connection was found between b and temperature t° , viz., $\log b = \alpha + \beta t$, α and β being constants. Combining the two logarithmic equations,—

$$\log q = a - y, \text{ if } \log y = \alpha + \log V + \beta t.$$

From this expression the amount of ammonia left in a solution after a certain volume of air has been drawn through it can be at once calculated, provided that, at the beginning of the aspiration, the strength of the solution was not much greater than that mentioned above.

DISCUSSION.

The PRESIDENT remarked that the subject investigated by the author was a very complicated one. He thought that the variations in the tension of the ammonia solution used in the experiments should be determined, as a valuable addition to the paper. It was probable that the results would be expressed by the equation— $\log p = A - B/T$ —where A and B are constants and T is the absolute temperature, which was applicable to many similar cases.

Dr. PERMAN, in reply to a question by Mr. Groves, stated that the conditions of volatilisation of ammonia from the solution had been varied by using flasks of different capacities and tubes of different diameters. In reply to Dr. Wilderman, he said that he considered that his results confirmed Henry's law for ammonia in dilute solution. In reply to the President, the author said he had found approximately the vapour-pressure of the ammonia solution used in most of the experiments at a temperature of 20°; it was about 70 m.m., although he had not found the vapour-pressure of solutions of different strengths, but intended to do so. He regarded the method of expressing the results given in his paper as the most convenient for the present, though as yet it was only empirical.

11. "On the Dissociation of Platinichloride in Dilute Solution; and the Production of Platinum Monochloride." By E. SONSTADT.

A solution of potassium platinichloride in 1000 parts of water undergoes no sensible change on heating for an hour or two. But a solution in 10,000 parts of water becomes turbid almost immediately on heating, and the turbidity increases until, after some hours, the liquid becomes nearly opaque. On continuing the heating for some days, water being added from time to time to maintain the bulk, a sediment forms and the liquid partially clears. By increasing the quantity of water during the heating, the clearance is facilitated. The reactions that take place are, first, the dissociation of the potassium platinichloride;



and then the decomposition of the platonic chloride, with formation of platinum monochloride ($PtCl$ or Pt_2Cl_3), hydrochloric acid, and hydrogen peroxide, thus:—



The yellow non-crystalline precipitate of hydrated platinum monochloride is difficult to collect, for it passes through the filter until the pores are clogged, and then the filtration is exceedingly slow. The precipitate should not be dried on the filter, but washed off, as far as possible, into a dish, and the water evaporated. When dried on the water-bath, the salt is brown; it still retains water, the last traces of which can scarcely be expelled before incipient decomposition begins; the anhydrous salt is black, or nearly so.

The clear filtrate, when concentrated, is strongly acid, and on drying and dissolving the residue in a little water, potassium chloride may be crystallised out. There is also a small proportion of potassium platinichloride, which may be either a portion of the original salt that has escaped decomposition, or may have been formed by a reverse action due to solution of the monochloride in the hydrochloric acid set free as described, in presence of hydrogen peroxide and air.

The hydrated salt, when treated on the water-bath with strong soda solution, turns brown, and dissolves to a slight extent. But the brown residue is only partly dehydrated, and recovers its original colour after washing and exposure. The soda solution deposits the unchanged salt on dilution and long exposure to the air. It dissolves readily in hydrochloric acid; slightly in hot dilute sulphuric acid, apparently without decomposition; in moderately dilute nitric acid, used in large proportion, it dissolves to a deep brown liquid, which, evaporated to dryness on the water-bath until no acid odour is perceptible, leaves a dark brown residue. This dissolves in hot water to a clear dark brown liquid, which, on further heating, suddenly deposits the whole of the original salt, less any impurities present, which remain in the solution. The precipitate, when collected on a filter, is deeper coloured than before, being of an orange tint. The filtrate is free from platinum; but on continued washing with water, the salt dissolves slightly, and the filtered liquid becomes clouded.

Advantage was taken of this process of purification to obtain the monochloride for analysis. An attempt to analyse about 0.1 gm. of the salt partially failed, owing to the presence of impurities taken up from the enormous proportion of distilled water used in its preparation. The monochloride, purified as described, was washed into a dish, and after drying on the water-bath, was transferred to a small porcelain crucible. The salt weighed 0.2575 gm. The crucible was cautiously heated, with frequent application of the cold lid. Traces of nitric acid came off as well as water. When moisture ceased to be given off, the salt weighed 0.2315 gm. It was then more strongly heated, when chlorine came off, and a final trace of water. The heat was then raised until the decomposition was complete. The platinum obtained weighed 0.1945 gm., which corresponds to 0.229 PtCl, a result which may be considered fairly satisfactory.

It might be supposed that platinum chloride itself, instead of dissociating from potassium chloride as in the described process, would be preferable, when it is desired to obtain platinum monochloride. But the presence of free acid in the best-dried specimens of platinum chloride renders it unfit for the purpose. A specially dried specimen of platinum chloride dissolved in 10,000 parts of water gave a barely perceptible reaction. With 15,000 parts of water, after heating for some hours, the liquid became turbid, and gave a slight deposit, but not equivalent to that obtainable from a corresponding solution of potassium platinum chloride. If, however, a solution of platinum chloride be evaporated on the water-bath with nitric acid, the residue dissolved in water, and the solution evaporated again two or three times, the platinum chloride is not appreciably decomposed, but retains a little nitric acid instead of the hydrochloric acid with which it is otherwise associated. A solution of platinum chloride thus prepared, heated in 5000 parts of water, gives a distinct turbidity, but the reaction is more nearly complete with double that proportion of water. The precipitate differs slightly in its properties from that obtained from the potassium salt; it is of a deeper tint, and is but slightly soluble in nitric acid. When treated with nitric acid and the solution evaporated over the undissolved portion, a weak solution only is obtained on adding water, which, on concentrating, deposits the salt, but not instantaneously. After evaporating the aqueous solution, the hydrate of platinum monochloride is insoluble in water, except, as in the former case, on continued washing. A specimen of the hydrate thus obtained weighed after drying on the water-bath, 0.1700 gm.; when anhydrous, it weighed 0.1549 gm., and after expulsion of the chlorine by ignition 0.1316 gm. of platinum remained. Theory requires from this quantity of platinum 0.1552 gm. PtCl.

The potassium platinum chloride used in the experiments had been several times re-crystallised, and was perfectly free from acid. The yield of platinum mono-

chloride was about five-eighths of that theoretically obtainable.

The author has made some experiments with the view of directly proving the presence of hydrogen peroxide in the liquid, though, considering the conditions of dilution and temperature, the quantity present at any time must be exceedingly small—the total amount produced according to the equation, assuming the reaction to be complete, being in the proportion of from 1 : 100,000 to 1 : 150,000 to the liquid present. In a solution that had been heated for five days, the author, using the chromic acid and ether test, obtained a faint colouration, hardly visible except to an observer of great sensitiveness. The author then prepared a solution in about 15,000 parts of water of some very pure crystals of potassium platinum chloride (a previous trial with a salt of only ordinary purity having failed), and heated the solution for several hours at a temperature slightly below 70°. There was only a slight turbidity, for the temperature was too low for good progress. On testing the solution the next day, a faint bluish tint in the ether was perceptible. That hydrochloric acid is a product of the reaction was proved as follows:—A hot strong solution of potassium platinum chloride was poured into a large quantity of a nearly saturated solution of pure potassium chloride. The precipitated platinum chloride was collected, well washed with water, and afterwards with alcohol, and dried on the water-bath. A solution of about one part of this salt to 10,000 of water was heated for five days, the precipitate allowed to settle, and filtered off. A portion of the solution was concentrated in an open dish to about one-fifth of its bulk, and was then distilled. Silver nitrate added to the distillate gave a precipitate of silver chloride insoluble in nitric acid.

12. "Effect of the Mono-, Di-, and Tri-chloracetyl Groups on the Rotatory Power of Methyl and Ethylic Glycolates and Tartrates." By PERCY FRANKLAND, F.R.S., and THOMAS STEWART PATTERSON, Ph.D.

With a view of ascertaining the rotatory effect of the halogens when attached at a point in the molecule remote from the asymmetric carbon atom, the authors have introduced the mono-, di-, and tri-chloracetyl groups into the methyl and ethylic tartrates and glycolates. This was effected by adding in each case with the acid chloride on the ethereal tartrate and glycolate respectively. The acid chlorides were in all cases prepared by heating the halogen-substituted acetic acid with phosphoric anhydride in a current of hydrochloric acid. Two acetyl groups were in all cases introduced, excepting in that of trichloracetyl, of which only one equivalent could be made to substitute in methyl and ethylic tartrates, whilst two equivalents reacted with the corresponding glycolates.

The rotations were in all cases determined at several different temperatures, so as to ascertain the influence of the latter.

The results are summarised in the accompanying table.

Methyl and ethylic di-monochloracetyl tartrates have also been prepared by Freundler (*Bull. Soc. Chim.*, 1895, [3], xiii., 1055—1063), who only obtained them as liquids with the following rotatory powers:—

Methyl di-monochloracetyl tartrate $[\alpha]_D^{18} = +3.5^\circ$

Ethylic " " $[\alpha]_D^{15} = +9.4^\circ$

In consequence of this discrepancy between Freundler's results and those of the authors, these two compounds have been prepared again (see next abstract), with the result that the authors' figures have been confirmed.

13. "The Rotation of Ethylic and Methyl Di-monochloracetyl tartrates." By PERCY FRANKLAND, F.R.S., and ANDREW TURNBULL, Ph.D.

The ethylic compound has been again twice prepared by the authors, using different proportions of monochloracetyl chloride and ethylic tartrate:—

First preparation, $[\alpha]_D^{20} = +7.90^\circ$

Second " $[\alpha]_D^{20} = +7.76^\circ$

	$[\alpha]_D^{15^\circ}$	$[\alpha]_D^{100^\circ}$	
Methylic glycerate	-4.80°	-8.31°*	(calculated).
" diacetyl glycerate	-12.04	-19.24°	(calculated).
" di-monochloracetyl glycerate ..	-12.91	-17.99	b. p. 197° (15 m.m.); m. p. 43-44°.
" di-dichloracetyl glycerate ..	-13.96	-17.18	b. p. 207° (20 m.m.).
" di-trichloracetyl glycerate ..	-14.20	-15.30	b. p. 199-200° (15 m.m.).
Ethylic glycerate	-9.18	-12.55°	(calculated).
" diacetyl glycerate	-16.31	-23.09°	(calculated).
" di-monochloracetyl glycerate ..	-16.80	-22.08	b. p. 198° (15 m.m.).
" di-dichloracetyl glycerate ..	-18.20	-21.10	b. p. 203° (15 m.m.).
" di-trichloracetyl glycerate ..	-18.70	-18.40	b. p. 202° (15 m.m.).
Methylic tartrate	+2.14°	+5.99°	m. p. 48°.
" diacetyl tartrate	—†	—	m. p. 103°.
" di-monochloracetyl tartrate ..	-0.62	+2.57	b. p. 217° (18 m.m.); m. p. 55°.
" di-dichloracetyl tartrate ..	+11.9	+10.9	b. p. 220-221° (15 m.m.); m. p. 64-65°.
" mono-trichloracetyl tartrate ..	+8.4	+10.15	m. p. 79-80°.
Ethylic tartrate	+7.66	+13.29	
" diacetyl tartrate	—†	—	m. p. 66.5°.
" di-monochloracetyl tartrate§ ..	+7.67	+11.81	b. p. 217° (15 m.m.); m. p. 27°.
" di-dichloracetyl tartrate ..	+16.3	+17.08	b. p. 225° (15 m.m.).
" mono-trichloracetyl tartrate ..	+15.5	+17.6	b. p. 185° (16 m.m.).

* These values for $[\alpha]_D$ at 100° for methylic and ethylic glycerates and diacetyl glycerates have been calculated from the data given in a paper by P. Frankland and MacGregor (*Trans.*, 1894, lxx., 768).

† $[\alpha]_D^{25^\circ} = -15.1^\circ$ (in absolute alcoholic solution).

‡ $n_D = +5^\circ$ ($t = 25, l = 1$).

§ See next abstract.

The substance subsequently crystallised (m. p. 27°), after which the rotation $[\alpha]_D^{20^\circ} = +7.61^\circ$ was found.

The methylic compound was also prepared and again gave the m. p. 55°; the rotation was $[\alpha]_D^{20^\circ} = -0.68^\circ$.

Thus the results obtained with both compounds by one of the authors (see previous abstract) were confirmed, whilst additional evidence of their accuracy was afforded by the ethylic compound being obtained in the crystalline state.

Ethylic mono-monochloracetyl tartrate in a state approaching purity was also prepared, and it was found to possess a considerably higher dextro-rotation than the corresponding di-acidyl compound, thus $[\alpha]_D^{20^\circ} = +11.44^\circ$.

It is, therefore, inferred that the higher figures obtained by Freundler may have been due to the presence of mono-acidyl compounds, and this is borne out by the lower densities and boiling-points which he records.

PHYSICAL SOCIETY.

Annual General Meeting, February 11th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

THE Report of the Council was read by Mr. Elder.

Dr. ATKINSON then presented the Treasurer's Report, and informed the Society of the improved condition of its finances. The difficulties of the previous year had arisen from the expenses incurred by the publication of Abstracts of current scientific literature; these difficulties had been surmounted without drawing upon the Reserve Fund. Very few Fellows had objected to the increase of subscription. In acknowledgment and appreciation of the Abstracts, now presented to all Fellows, many of the original Life-members had lately made additional voluntary donations to the funds of the Society, thus sharing with new Fellows the extra outlay involved by the Abstracts. It was to be hoped that all Life-members would adopt this course, more especially as the scope of scientific literature covered by the Abstracts was now being extended to British as well as to foreign sources.

Votes of thanks were passed to the Council, the Officers, and to the Council of the Chemical Society for the use of their rooms at Burlington House.

Two Honorary Fellows were unanimously elected by ballot, *i.e.*, Riccardo Felici, Professor in the University of Pisa; and Emelio Villari, Professor in the University of Naples.

Council and Officers for the forthcoming year were elected as follows:—

President—Mr. Shelford Bidwell.

Vice-Presidents, who have filled the Office of President

—Dr. J. H. Gladstone, Prof. G. C. Foster, Prof. W. G. Adams, The Lord Kelvin, Prof. R. B. Clifton, Prof. A. W. Reinold, Prof. W. E. Ayrton, Prof. G. F. Fitzgerald, Prof. A. W. Rücker, Capt. W. de W. Abney.

Vice-Presidents—Prof. C. Vernon Boys, Maj.-Gen. E. R. Feasting, Mr. G. Griffith, Prof. J. Perry.

Secretary—Mr. H. M. Elder, 50, City Road, E.C.

Foreign Secretary—Prof. S. P. Thompson.

Treasurer—Dr. E. Atkinson.

Librarian—Mr. W. Watson.

Other Members of Council—Prof. H. E. Armstrong, Mr. Walter Baily, Mr. L. Clark, Dr. A. H. Fison, Mr. R. T. Glazebrook, Prof. A. Gray, Prof. J. Viriamu Jones, Mr. S. Lupton, Prof. G. M. Minchin, Mr. J. Walker.

THE PRESIDENT then read an Address, in which the aims and history of the Physical Society were outlined. This Address will be published in full in due course.

Prof. RÜCKER said that, among the new and useful departures lately made by the Physical Society, the institution of a Presidential Address was particularly worthy of notice; it was very desirable, from time to time, to hear a summary of what had been achieved during the year; it was also desirable that the objects of the Society should be, from time to time, definitely stated: this departure had been fully justified by the Address of Mr. Shelford Bidwell.

A paper by Mr. G. H. BRYAN, on "*Electro-magnetic Induction in Plane, Cylindrical, and Spherical Current Sheets, and its Representation by Moving Trails of Images*," was read by Mr. ELDER.

The phenomena of induction in a cylindrical conducting

sheet in a two-dimensional field, and of induction in a spherical sheet in any field due to the generation or motion of poles, magnets, or currents, in the presence of the sheet, can be represented by moving trails of images which are but slightly more complicated than the well-known trails of images in a plane sheet. The images, representing the potentials of the induced currents on the two sides, start from the source of disturbance and its inverse point, and move normally away from the surface of the sphere and cylinder, with velocity varying directly as the disturbance. At the surface of the sheet this velocity becomes equal to the corresponding velocity for a plane sheet. The images are in most cases similar in nature to the inducing source of disturbance, and their intensities are found, in every case, to vary as a power of the distance from the centre. The images due to the sudden generation of a magnetic pole in the presence of a spherical sheet are, however, analogous to the hydro-dynamical image of a source in a sphere.

Dr. S. P. THOMPSON said the method and the results obtained would find useful application in the solution of many allied problems.

The PRESIDENT proposed a vote of thanks to the author. The meeting was then adjourned until Saturday, February 26th, on which occasion the Physical Society will visit Eton College.

Fellows are informed that a train leaves Paddington for Windsor at 2.25 p.m.; this arrives in time for the meeting, which is at 4 p.m.

ROYAL INSTITUTION.

General Monthly Meeting, February 7th, 1898.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

THE following were elected Members:—E. Cortes, J. S. Fairfax, S. Fisher, G. Humphreys-Davies, O. Inray, J. W. Jarvis, J. Levinstein, J. S. MacArthur, The Right Hon. Lord Monk Bretton, F. J. Quick, E. Riston, C. Samson, Mrs. R. L. Smith, and Rear-Admiral A. K. Wilson, V.C., C.B.

The special thanks of the Members were returned to Mrs. Tyndall for her liberal donation of £1000, presented in the name of the late Dr. John Tyndall, D.C.L., F.R.S., for the Promotion of Science. Thanks were also returned to Sir Frederick Abel, Sir Andrew Noble, and Professor Dewar for donations to the Fund for the Promotion of Experimental Research at Low Temperatures.

It was announced that the Centenary of the Royal Institution would be celebrated next year.

EDINBURGH UNIVERSITY CHEMICAL SOCIETY.

Monday, January 31st, 1898.

Dr. BOLAM in the Chair.

DR. MACDONALD read a paper discussing the Hydrogenation of Terephthalic and Phthalic Acids, by v. Baeyer, and of Naphthols and Naphthylamines by Bamberger.

The work of these investigators shows that the characteristic properties of aromatic compounds are due to the symmetry of the double bonds in the molecule. As soon as a ring loses this symmetry by hydrogenation, it ceases to possess aromatic and assumes fatty properties. Baeyer's results are of unique importance in showing that maximum stability is conditioned by a symmetrical disposition of the double bonds in the molecule. Thus the dihydroterephthalic acids $\Delta^2,5$ and $\Delta^1,3$ readily pass into $\Delta^1,4$, the most symmetrical form. Again, the most stable dihydrophthalic acid, $\Delta^2,6$, is the most symmetrical;

and the least symmetrical hydrophthalic acids, $\Delta^2,3$, $\Delta^1,3$, and Δ^3 , are either incapable of existence or exist only under the most favourable conditions.

In the light of the easy transposition of these compounds and from considerations of energy and elasticity and perfect symmetry of the benzene molecule, we could not expect that two ortho-di-derivatives of benzene could possibly exist, at least under any but the most extraordinary conditions.

Dr. DOBBIN then read a short review of Professor Oscar Loew's book entitled "The Energy of Living Protoplasm" (a review of this book has already appeared in the CHEMICAL NEWS, vol. lxxiv., p. 255). His primary object in doing so was to call the attention of the members to the existence of the book, as it did not appear to be so well known, at least to chemists, as its importance to them, as well as to physiologists and biologists, would seem to deserve. In his book the author attacked, from a purely chemical point of view, the problem of the differences in character and properties of living as contrasted with dead protoplasm, and endeavoured to refer these differences to certain atomic rearrangements (which cannot be more minutely specified here) taking place within the albumin molecules of the protoplasm. Although he did not in any way commit himself to defending the author's views, the speaker strongly recommended the book to the attention of the members, as an interesting and eminently suggestive one.

CORRESPONDENCE.

CAMPHOR AND ITS DERIVATIVES.

To the Editor of the Chemical News.

SIR,—In a letter addressed to you by Prof. Noyes (CHEM. NEWS, vol. lxxvii., p. 70), your correspondent argues against one of two formulæ for camphor which he represents as introduced by me during a discussion at the Chemical Society.

May I remove an obvious misunderstanding on the part of Professor Noyes?

At the meeting in question a new formula for camphoric acid was employed by Drs. Crossley and Perkin. I pointed out that this would seem to necessitate the representation of camphor by one or other of two formulæ, of which one might be regarded as the more probable; but I added that "before either could be accepted, an explanation must be furnished of the changes involved in the production of such compounds as cymene and carvacrol from camphor—changes which, it must be remembered, are readily explained by Bredt's formula" (see CHEMICAL NEWS, vol. lxxvi., p. 297).

I do not prefer either of these formulæ to that of Bredt, which, in common with many chemists, I regard as the most satisfactory formula for camphor yet devised.—I am, &c.,

M. O. FORSTER.

The Royal College of Science, London,
South Kensington, S.W.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Seances, de l'Académie des Sciences. Vol. cxxvi., No. 4, January 24, 1898.

On an Interferential Spectroscope.—Ch. Fabry and A. Perot.—In a previous communication the authors have

referred to a new method of interferential spectroscopy; they now give more details of the arrangement of the apparatus. The method consists of observing the rings, localised to infinity, produced by the transmission through a film of air compressed between two plane surfaces of silvered glass. The two surfaces should be brought into perfect parallelism, and one of them must be fitted so that it can be moved away from the other, still retaining its parallel position, up to several centimetres. A sheet of glass silvered on each side may be used with excellent results, but in this case the thickness cannot be varied.

Conditions under which the Formation of the Alkaline Carbides, the Alkaline-earthly Carbides, and Carbide of Magnesium take place.—Henri Moissan.—This article will be inserted in full.

Separation of Thorium and the Cerite Earths.—G. Wyruboff and A. Verneuil.—This paper will be inserted in full.

Hydramides and Isomeric Bases (Glyoxalidines).—Marcel Delépine.—The heat of formation of anisic aldehyd, $C_6H_4(CHO)(OCH_3)_2$, was found to be +63.1 cal.; that of anishydramine, $(C_6H_5O)_2N_2$, 48.4 cal.; that of anisic, $C_{10}H_{12}N_2O_3$, 64.6 cal.; that of furfuramide, $(C_5H_4O)_3N_2$, +0.35 cal.; that of furfurine, $C_{15}H_{12}N_2O_3$, 17.9 cal.; and that of chlorhydrate of furfurine, $C_{15}H_{12}N_2O_3.HCl$, +28.7 cal.

Researches on Ouabaine.—M. Arnaud.—The author has prepared a number of compounds and derivatives of this body which he hopes will help to define it from the chemical point of view. At least three different crystalline hydrates have been obtained by evaporating at temperatures of 10° to 20°, about 30°, and about 60°. The first contains 17.5 per cent of water when dried in the desiccator over sulphuric acid, the second 11.2 per cent, and the third 9 per cent. The rotatory power of ouabaine in a 1 per cent aqueous solution is $[\alpha]_D = -30.6^\circ$.

Synthesis of Terebic Acid.—E. E. Blaise.—Terebic acid obtained by the method described at length by the author melts at 174°; its identification is made complete by its transformation into methyl-2-pentanolate, which is done by two consecutive slow distillations, and then warming for some minutes with sulphuric acid and water (2—1); this is neutralised with carbonate of soda and exhausted with ether. The alide thus produced boils at 206—207°.

Manufacture of Oil of Acetone, and especially Methyl ethylacetone, from the Wash-waters from Wool Cleaning.—A. and P. Buisine.—The volatile fatty acids isolated from these wash-waters are saturated with lime and the solution evaporated to dryness. The dried salts of lime are then distilled. The return of oil of acetone from waters of 11° Baumé is, by this method, about 15 litres per cubic metre, and the operation can be carried out at a very low price.

Anhydrous Sulphate of Lime produced by the Complete Dehydration of Gypsum.—A. Lacroix.—The object of this paper is to show that, by the complete dehydration of gypsum, a crystallised sulphate of lime is formed which is different to *anhydrite*. Crystals of gypsum were slowly heated in an oil-oven to 145°; they could then be polished as easily as before. Examination with polarised light shows that a dimorphous sulphate of lime is formed, probably triclinical in its crystalline form; both its refrangibility and its bi-refrangibility are much lower than those of *anhydrite*.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 23.

Remarks relative to the Note presented by R. Franchot entitled "On Nascent Hydrogen."—D. Tommasi.—The author asserts that M. Franchot has ignored his work on the subject of nascent hydrogen,

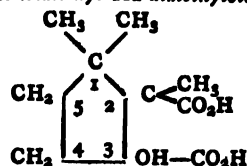
carried out in 1877, and published in the *Comptes Rendus de l'Institut des Sciences de Milan*, 1877 and 1878.

On the Thermochemical Theory of a Battery with Carbon Electrodes.—D. Tommasi.—The author considers as inadmissible the theory that, in an electric battery with carbon electrodes such as his, the disengagement of electricity is entirely due to the chemical action of the chloride of sodium on the peroxide of lead surrounding one of the carbons—firstly, because in all voltaic couples in which the solution of chloride of sodium is used as the electrolyte, it is always the water that is decomposed, and never the chloride of sodium; and, secondly, because peroxide of lead has no action even on concentrated chloride of sodium.

Basic Salts of Magnesium.—M. Tasally.—Oxychlorides of magnesium have long engaged the attention of chemists, though neither the oxybromide nor the oxyiodide had been prepared: the author has now succeeded in obtaining an oxybromide, though all his efforts to prepare an oxyiodide have been in vain. Three hundred grms. of water, containing 145 grms. of crystallised bromide of magnesium, were heated to boiling; the flame was withdrawn, and 5 grms. of calcined magnesia was added in small portions. It is again boiled, and the magnesia dissolves; towards the end of the operation the temperature is raised to 150°. The solution is filtered hot, and placed in a well-stoppered bottle. After a fortnight the oxybromide is deposited in small crystalline needles, acting on polarised light, showing longitudinal extinctions. The composition of this body answers to the formula $MgBr_{2.3}MgO_{1.2}H_2O$. When heated to 120° in dry air it loses half its water. These oxybromides undergo a change when exposed to the air, with the fixation of carbonic acid; they are decomposed by water, alcohol, and most reagents.

Syntheses by means of Tetrachloride of Phthalyl melting at 88°. Part I.—The Study of some Homologues of Diphenylanthrone.—A. Guyot.—A very long paper, not suitable for abstraction.

The Constitution of Camphoric Acid.—L. Bouveault.—Camphoric acid is bibasic, and has many points of resemblance with succinic and phthalic acids. It constitutes the *cyclo-pentane-trimethyl-112-dimethyl-23*,—



On the Schiff Reaction applied to some Substituted Fuchsines.—P. Cazeneuve.—Certain homologues of fuchsine, such as orthotolylfuchsine and xylylfuchsine, are very sensitive to the action of aldehyds, after decolouration by SO_2 . The colour obtained is bluer than with ordinary fuchsine.

The Congealing-point of Milk and some Allied Facts: Reply to Various Objections.—J. Winter.—This note is a *résumé* of a communication made by the author to the last meeting of the Society, in July, 1897, with the object of replying to several criticisms which had been passed on his work on this subject.

MISCELLANEOUS.

International Health Exposition.—This Exhibition will be held in the Industrial Building, New York, from April 25th to May 31st, 1898, under the supervision of Mr. Chas. F. Wingate. It will cover everything relating to health both in-doors and out-of-doors, and will show the progress made in sanitary matters during the century. An Educational Congress will also be held in connection

with the Exhibition, and a series of popular lectures will be provided every day during the time it is open. Exhibitors having appliances, or systems, which show a genuine advance, will be encouraged to provide lecturers or demonstrators themselves. The Exhibition will be divided into two principal departments, viz., Domestic Sanitation and Municipal Hygiene, these again being further subdivided into groups and sections.

MEETINGS FOR THE WEEK.

- MONDAY, 21st.**—Society of Arts, 8. (Cantor Lectures). "The Principles of Design in Form," by Hugh Stannus, F.R.I.B.A.
- TUESDAY, 22nd.**—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
- Society of Arts, 8. "The Regalia of England," by Cyril Davenport.
- WEDNESDAY, 23rd.**—Society of Arts, 8. "Children's Sight," by R. Brudenell Carter, F.R.C.S.
- THURSDAY, 24th.**—Royal Institution, 3. "Some Italian Pictures at the National Gallery," by Jean Paul Richter.
- FRIDAY, 25th.**—Royal Institution, 3. "The Theory of Colour Vision applied to Modern Colour Photography," by Capt. Abney, C.B., D.C.L., F.R.S.
- SATURDAY, 26th.**—Royal Institution, 3. "The Structure of Instrumental Music" (with Musical Illustrations), by William H.adow, M.A., B.Mus.
- Physical, 4. (Instead of Friday, Feb. 25. In the Chemistry Lecture Room, East's Lane, Eton College). The Rev. T. C. Porter will describe:—
1. A New Theory of Geysers. 2. A New Method of Viewing Newton's Rings. 3. Experiments bearing on the Sensation of Light. 4. A Method of Viewing Lantern Projections in Stereoscopic Relief. 5. Winter Observations on the Shadow of El Teide, with a New Method for Measuring approximately the Diameter of the Earth. 6. Temperature of the Water of Niagara.

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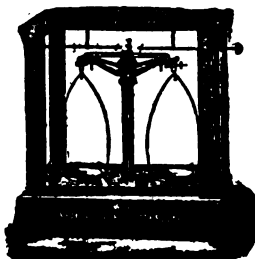
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CONTENTS.

ARTICLES:—	PAGE
Conditions under which the Formation of the Alkaline Carbides, the Alkaline-earthly Carbides, and Carbide of Magnesium take place, by M. Henri Moissan	85
Some Remarks on the Production of Steel Castings, by Sergius Kera, M.E.	86
On Chemically Inactive Elements, by Dr. Joachim Sperber	87
The Kekulé Memorial	88
On the Dissociation Spectra of Melted Salts—Sulphur, Phosphorus, and the Solid Phosphorous Compounds, by A. de Gramont	88
London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.	90
Salts of Dinitro-alpha-naphthol with various Metallic Bases, by T. H. Norton and H. Lowenstein	90
The Relation of the Taste of Acids to their Degree of Dissociation, by Theodore William Richards	91
The Electrolytic Determination of Cadmium, by Daniel L. Wallace and Edgar F. Smith	93
Testing of Formaldehyd, by Carl E. Smith	94
NOTICES OF BOOKS	95
CHEMICAL NOTICES FROM FOREIGN SOURCES	95
MISCELLANEOUS	96
MEETINGS FOR THE WEEK	96

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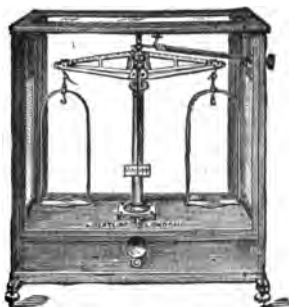
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VOL. LXXVII., No. 1996.

CONDITIONS UNDER WHICH THE FORMATION OF THE ALKALINE CARBIDES, THE ALKALINE-EARTHY CARBIDES, AND CARBIDE OF MAGNESIUM TAKE PLACE.

By M. HENRI MOISSAN.

By the aid of the electric furnace we have been able to prepare a large number of definite crystallised carbides, by reducing the oxides or carbonates by means of carbon. Certain of these, however, such as the alkaline carbides, cannot be prepared in this manner. It seemed interesting to find out why this method of preparation cannot be applied to this series of compounds.

In his paper, on a new class of metallic radicals, M. Berthelot (*Annales de Ch. et de Ph.*, Series 4, vol. ix., p. 385, 1886) announced the existence of the compounds C_2HNa and C_2Na_2 , which were obtained by the action of a more or less elevated temperature on sodium, kept in an atmosphere of acetylene. The body C_2Na_2 thus formed is carbide of sodium, analogous by means of its collective properties to carbide of calcium, which I have prepared with ease in the electric furnace, and which has been the starting point of the acetylene industry. It seemed reasonable to imagine that the alkaline-earthly carbides, heated in an atmosphere of acetylene, might also form alkaline-earthly carbides. But the impossibility of obtaining these metals in a state of purity has prevented this experiment being carried out.

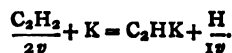
Carbide of Potassium.—M. Berthelot has prepared this compound by the action of acetylene at a dull red heat on the alkaline metal. When we heat a mixture of carbonate of potassium and carbon ($CO_3K_2 + 2C$) in the electric furnace, with a current of 900 amperes and 45 volts, we obtain a black pulverulent substance consisting of almost pure carbon, containing merely traces of the alkaline metal, and not producing any disengagement of gas on contact with water. Thus with a current of such intensity no carbide of potassium is formed. In a second experiment we heated the mixture of black flux and carbon, which is used for the preparation of potassium, in the electric furnace in a carbon tube closed at one end. The heating lasted eight minutes, and the current, weaker than before, was 45 volts and 350 amperes. After cooling, the product drawn from the tube was found to consist of a coarse powder, though some pieces had the appearance of mass, and of having been melted, thrown on water they immediately gave off gas. If this black substance be merely moistened with water it becomes incandescent, and gives off a gas burning with a reddish violet flame. Some fragments even produce a slight explosion. Treated with water in a tube filled with mercury it gives rise to a slight disengagement of gas. The black residue is formed of carbon, and the filtered water has taken a yellowish tint. This solution is very alkaline, and contains a small quantity of carbonate and of cyanide.

The gas given off consists of a little nitrogen, hydrogen, and acetylene. Two analyses of this gaseous mixture gave us 70 and 66·6 per cent of acetylene. The disengagement of gas is very slight, and the quantity of carbide of potassium produced under these conditions is very small. These first experiments show us that the temperature of the electric furnace is much too high to make the preparation of carbide of potassium possible by its use.

On the other hand, we have remarked that metallic potassium was slowly attacked in the cold at the ordinary

pressure by acetylene gas. If the experiment is sufficiently prolonged the attack may even be complete. In one experiment we made, we placed a fragment of potassium in the top of a tube, 0·5 metre in length, closed at its upper extremity. This tube was filled with acetylene gas, and placed in a mercury bath. From the second day it was noticed that the level of the mercury was rising sensibly. The absorption continued slowly, and the experiment was stopped forty-nine days later.

At the commencement, the apparatus contained 17 c.c. of acetylene gas at 0° and 760 m.m. pressure; after the experiment the volume remaining was 8·35 c.c., containing 4·48 c.c. of hydrogen (the whole being reduced to 0° and 760 m.m.). The volume of acetylene absorbed was 8·65 c.c. It is practically the double of that of the hydrogen produced, which may be explained by the following equation:—



We thus obtain in the cold a white product* adhering slightly to the tube, and which is decomposed by contact with cold water, giving pure acetylene. The volume of the gas obtained under these conditions was 7 c.c., which is slightly lower than the theoretical quantity. This slight difference is due to the polymerisation of a small portion of the acetylene. Thus in the cold, by the action of potassium on acetylene, we obtain the compound C_2HK , or potassic acetylene, already mentioned by M. Berthelot, an intermediary compound between acetylene C_2H_2 and carbide of potassium C_2K_2 .

Carbide of Sodium.—The reduction of carbonate of sodium by carbon, under the influence of an intense current (1000 amperes and 70 volts), did not produce a carbide. On the contrary, by heating in a closed crucible, placed in a carbon tube, a mixture of soda and sugar-charcoal, by means of a weak current (350 amperes and 45 volts), we obtained a black powder, which, on contact with cold water, gave a slight disengagement of acetylene. The absorption of acetylene gas by sodium, in the cold, is much slower than with potassium; it is, in fact, almost nil.†

On account of this very slight absorption, we modified the experiment in the following manner:—A sealed tube was prepared containing sodium and dry liquid acetylene, and left at the temperature of the laboratory. At first the action is slow, but after several days the sodium becomes tarnished and covered with a layer of a yellowish white colour, the thickness of which keeps constantly increasing. At last the transformation of the metal is entirely complete. The tube, cooled down to a very low temperature, was at length opened, and the gas collected by means of a mercury pump. It was easy to separate the free hydrogen from the excess of acetylene.

The product, of a yellowish white colour, which has replaced the sodium, was analysed in the following manner:—A given weight of the compound was decomposed by water, and the acetylene collected and measured; it was then analysed to make certain of its purity. The sodium is estimated by the alkalimetric titration of the soda formed. The derivative thus obtained in the cold corresponds to the formula C_2HNa . This sodic acetylene, again, is the intermediate compound between acetylene and the carbide.

It is not indispensable to use liquid acetylene in this

* The transparent compound, which is produced by the action of potassium on acetylene in the cold, at first appears to possess a blue colour in the portions which were in contact with the metal when examined through a lens. This tint disappears as the transformation of the potassium takes place. This phenomenon may be compared with the blue colouration taken by chloride of potassium at a red heat in the presence of vapour of potassium, mentioned by Le Roux (*Comptes Rendus*, vol. lxviii., p. 1022, 1868), and which seems to result from the diffusion of the metal.

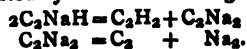
† The more easy formation of potassic acetylene explains why M. Berthelot met with traces of acetylene in the hydrogen produced by the decomposition of water on contact with potassium, while he found none in the hydrogen obtained by means of sodium.

operation; the same reaction can be carried out at the ordinary temperature with the compressed gas. The speed of the reaction, however, appears to be a function of the pressure. Thus, as we remarked above, the combination of sodium and acetylene is difficult to produce at the ordinary pressure, and it requires several months to become noticeable. At a pressure of one atmosphere the combination is more rapid, and if we heat slightly, even at a lower pressure, the attack proceeds with greater rapidity. Towards 50° it is already distinct, and we have succeeded in completely transforming a small fragment of sodium into the compound C_2HNa , by keeping it in contact with acetylene gas for fifteen days, at a temperature of 70° .

Dissociation of the Compound C_2HNa .—The white body having the formula C_2HNa , obtained by the action of acetylene upon sodium, in the cold, was placed in a tube of Bohemian glass closed at one end, and connected with a mercury pump. If this compound is slowly heated *in vacuo* we notice that it becomes brown, and at first gives off pure acetylene gas.

By continuing the heat the colour deepens, and, at the same time that the acetylene is given off, a small quantity of liquid carbides of hydrogen is condensed in the cold part of the tube. By then stopping the experiment, before spoiling the shape of the glass tube, the solid residue which remains gives off pure acetylene on contact with cold water: this is the carbide of sodium of M. Berthelot, having the formula C_2Na_2 ; if, instead of decomposing this compound with water, we continue to heat it *in vacuo* up to redness it will no longer give off any gas; black carbon only remains at the bottom of the tube,* and we notice a bright ring of metallic sodium condense above, in the cool part of the tube.

Thus, by elevating the temperature, *in vacuo*, to a point which need hardly reach the softening point of Bohemian glass, carbide of sodium is decomposed into carbon and sodium. This fact explains our want of success in our experiments on the reduction of soda, or of carbonate of sodium by carbon. The temperature of the electric furnace is much too high for carbide of sodium to be able to exist under these conditions. The preceding experiments may be represented by the two following equations:—



Carbide of Lithium.—We have already remarked, *à propos* of the preparation of carbide of lithium, that even with a current of 350 ampères and 50 volts, it is not necessary to heat for more than ten minutes, or else nothing but graphite remains at the bottom of the carbon tube in which the experiment was made. The temperature of this operation is of great importance from the point of view of the amount of carbide produced. This compound of lithium is in fact more easily dissociated by heat than is carbide of calcium. The temperature of decomposition of carbide of lithium is intermediate between that of carbide of sodium and that of the alkaline-earthly carbides.

Carbide of Calcium.—A certain number of experiments have shown us that, in the electric furnace, carbide of calcium can be decomposed if we make use of currents of too great intensity:—

1. In preparing small quantities of carbide of calcium with currents of 1200 ampères and 60 volts, it has happened, if the experiment lasts ten minutes, that we have obtained a residue of pulverulent graphite, containing only traces of carbide of calcium. The compound has not volatilised, for the carbon remains in the crucible. It is more reasonable to suppose that a dissociation of the carbide has taken place, the calcium distilling off with ease, leaving the pulverulent residue of carbon.

2. In the experiments made by means of a cold Deville tube, which were carried out with the object of

studying the condensation of the vapour of carbon, we never obtained any condensation of carbide of calcium. If, in fact, we heat melted carbide of calcium in the electric furnace, all we find on the copper tube, through which cold water is kept circulating, is graphitic dust, pulverulent lime, and calcium; this latter, on contact with water, gives off hydrogen, the purity of which has been verified by a eudiometric analysis. As with the carbides of sodium and lithium, but at a much higher temperature, carbide of calcium can be decomposed into the metal, and carbon.

Carbide of Magnesium.—By carefully heating powdered magnesium in a current of acetylene gas (a reaction suggested by M. Berthelot), we obtain an impure carbide of magnesium, mixed with carbon, but unmistakably giving acetylene gas on contact with cold water.

This carbide of magnesium was placed in a double crucible of pure graphite, and heated in a carbon tube closed at one end, using 600 ampères and 60 volts; the experiment lasted six minutes. After cooling, the black material taken from the bottom of the crucible gave no disengagement of gas on contact with water. The carbide of magnesium was totally decomposed under the influence of this high temperature. This experiment perhaps explains why we have been able to melt magnesia and bring it to quite a liquid state in carbon crucibles, without this oxide being in any way reduced. At the temperature of fused magnesia, carbide of magnesium can no longer exist.

Conclusions.—To sum up, by the action of cold acetylene gas, or by the action of liquid acetylene, with or without pressure, we can obtain the intermediate sodic and potassic acetylenes in a state of purity, C_2KH and C_2NaH . By elevating the temperature these bodies can be decomposed; they cause a disengagement of acetylene, and they leave as residue the carbides C_2K_2 and C_2Na_2 .

By a much greater elevation of temperature these carbides are dissociated into the metals and carbon. The phenomenon is identical with the carbides of the alkaline earths, though a much higher temperature is required. The same holds good with carbide of magnesium.

The stability of these carbides, under greater and greater variations of temperature, goes on increasing from the alkaline metals up to the alkaline-earthly metals.

These experiments have established the fact that the carbides of potassium, sodium, and of magnesium cannot be produced at the temperature of the electric furnace.—*Comptes Rendus*, cxvii., No. 4.

SOME REMARKS ON THE PRODUCTION OF STEEL CASTINGS.

By SERGIUS KERN, M.E., St. Petersburg.

In 1894 we introduced the crucible steel process for the production of steel castings at the New Admiralty, St. Petersburg. The coke steel furnace gives daily nearly 3 cwts. of steel; this quantity is the production of four crucibles per heat. An annealing furnace is attached to the small foundry; every casting being subjected to the annealing process in charcoal.

The moulding sand consists of about 90 per cent of fine quartz sand and 10 per cent of the best fire-clay. This mixture is mixed with a small quantity of sawdust and rye-flour. As a wash for mouldings, malt boiled with water is used.

We consider that ferro-silico-manganese is the most powerful agent as an addition to ready-made steel for the production of sound castings; the most useful alloy containing, on the average, 10 per cent of silicon and 15 per cent of manganese. Metallic aluminium is of second importance, especially if good raw materials are used; we prefer an addition of ferro-aluminium (14 per cent of aluminium) with the silico-manganese.

* We have shown, by experiment, that this powder was formed of pure carbon. It no longer gave off any gas on contact with water.

For steel castings the following raw materials are used:—Roundings coming from the punching of soft steel ship-plates for rivet holes, and soft puddled iron. The plates contain on the average 0.4 per cent of manganese, and such material is suitable—as we remarked that steel castings containing some 0.6 per cent of manganese, even if annealed, are somewhat dry and of a crumbling nature.

Once a month the chemical composition of steel castings is ascertained. In the ordinary run, the composition of various castings for ship-building is as follows:—

Carbon	0.40 per cent.
Manganese	0.42 "
Silicon	0.35 "
Sulphur	0.02 "
Phosphorus	0.04 "

The mechanical tests on pieces half an inch in diameter and four inches in length give:—21 tons per square inch as elastic limit, 45 tons per square inch as resistance, with an elongation of 8 to 10 per cent.

In case of castings to resist water or steam pressure, we prefer the use of steel stronger in carbon, and have 0.6 per cent of carbon in the metal. Such a metal we recommend for the different pieces of Belleville boilers, mostly hollow and of capricious forms.

The crucible charge for ordinary castings contains:—

Steel plate roundings	50 pounds.
Steel plate shearings	10 "
Puddled iron	20 "

When the charge is melted, which results after some five hours, an addition is made, consisting of—

Silico-manganese	0.75 of a pound.
Ferro-aluminium	0.15 "
Ferro-manganese (containing 80 per cent Mn)	0.10 "

In total, the addition weighs 1 pound, and is introduced into each crucible by means of a funnel, and remains there for twenty minutes. After this lapse of time the crucibles are taken out, and the casting commenced. Some minutes after, the moulds are opened, the castings released, and covered again with the moulding sand. In such a state they cool, being finally introduced into the annealing furnace.

The crucible charge for water-tight castings consists of—

Ship-plate roundings	55 pounds.
Ship-plate shearings	10 "
Puddled iron	12 "
Refined best cast-iron	3 "

The following is the addition for such a steel:—

Silico-manganese	0.70 of a pound.
Ferro-aluminium	0.25 "
Ferro-manganese (containing 80 per cent Mn)	0.15 "

This addition, before the casting, remains in the crucible for twenty-five minutes.

The refined cast-iron used in the charge is very pure, coming from charcoal blast-furnaces; and after refining, which is done by re-melting the material in crucibles, contains:—

Total carbon	3.78 per cent.
Manganese	0.58 "
Silicon	0.28 "
Phosphorus }	0.03 "
Sulphur .. }	

There is a good field here for private works of steel castings. The big establishments make castings for their own use only—having no time for outside orders,—and the one or two small steel foundries work still empirically, sending out good and bad material. We had in hand steel castings sent to the Admiralty and rejected; they contained 0.75 per cent of carbon.

February 14, 1898.

ON CHEMICALLY INACTIVE ELEMENTS.

By Dr. JOACHIM SPERBER.

INASMUCH as the elements, with the exception of hydrogen as unity, act with a force or valency which is not in proportion to their atomic weights, I drew the following conclusions, based on the doctrine of the parallelogram of forces:—

1. That atoms enter into reaction at definite angles in such a manner that the valency only indicates the value of the component which the atom contributes, in the direction of the molecular movement in the resulting compound.

2. That the valency of an element (V) equals the atomic weight (a) into the cosine of the angle (φ), at which the atom enters into combination.

$$V = a \cos. \phi.$$

This angle will in some instances depend on the direction of the vibrations of the atom itself within the molecule; in other instances, in the direction of the vibration of the molecule itself. For—

$$\begin{aligned} \phi &= 90^\circ \\ \cos. \phi &= 0 \\ V &= a.0 = 0; \end{aligned}$$

that is, if an atom vibrates at right angles to the molecular vibrations—as, for instance, in the case of an atom with transverse vibrations in the presence of gases with their strong tendency to longitudinal vibrations—it has no valency, therefore no affinity, and consequently cannot enter into chemical combination.

My theory of valency, therefore, presumes the existence of elements that cannot enter into chemical combination, because their atoms have a prevailing tendency to transverse vibrations; there is, too, substantial proof of this, for the cosmic ether is without doubt such an element, since, as is known, it does not enter into chemical combination, and its vibrations are transversal. Moreover, the chemically inactive elements, argon and helium, have recently been discovered; and if they cannot be made to enter into chemical combination,—as up to the present is the case, in spite of zealous endeavours in that direction,—then, in accordance with my theory of valency, this is to be attributed to a prevailing tendency on the part of their atoms to vibrate transversally, which brings these elements into close relationship to the cosmic ether, perhaps to be regarded as condensation products of different grades.

It is important to confirm this, for when once the cause of the inactivity of argon and helium is known, the sooner will a means be discovered for rendering them active.

We know from experience that atoms of elements have less affinity among themselves than towards other atoms; the greater activity of nascent elements is, indeed, due to this fact. When, therefore, the atoms of an element show no affinity towards other atoms, they can have no affinity among themselves. Hence it follows that the inactive elements must be monatomic; this likewise agrees with all we know up to the present about argon and helium.

International Photographic Exhibition at the Crystal Palace, 1898.—H.R.H. The Prince of Wales has graciously consented to open the Exhibition. Intending exhibitors are asked to note that the date of opening of the Exhibition by His Royal Highness has been fixed by him for Monday, April 25th, and not Wednesday, April 27th, as originally announced. The latest date for the reception of exhibits in each Section will therefore be two days earlier than that first stated on the prospectus.

* This presumes components at right-angles, as are spontaneously and frequently engendered in Nature.

THE KEKULÉ MEMORIAL.

THE death of August Kekulé on February 13, 1897, terminated a career rich in scientific achievement. In him we have lost an investigator who has exerted a profound influence on the development of chemistry.

The theory of valency and of the linking of atoms, and our present views as to the structure of carbon compounds, have acquired their definite form and clearness by the labours of Kekulé. His theory of benzene derivatives in particular has given the most powerful impulse to investigation in the domain of pure chemistry; while no scientific theory has done more to promote the development of chemistry as a branch of industry.

While Kekulé is eminent by his scientific achievements, he is not less so by reason of the effects produced by his teaching. The publication of his "Lehrbuch der Organischen Chemie" marked an epoch in the history of chemistry. This treatise has done more to familiarise chemists with modern views than any other work of the kind.

The greater number of German professors of chemistry, and many of those in other countries, have either studied under Kekulé or under those who were his pupils; and gratitude calls for some permanent memorial of his striking personality.

Such a memorial would be a statue in bronze of the founder of structural chemistry, which would be fitly placed in front of the Chemical Institute at Bonn—in the place where, for thirty years, he lived, and taught, and worked.

All friends, admirers, and pupils of Kekulé are accordingly invited to contribute to this object. Subscriptions will be forwarded to the Central Committee at Bonn, and will be received by Dr. Hugo Müller, 13, Park Square East, Regent's Park, N.W.

JAMES DEWAR,
G. CAREY FOSTER,
T. E. THORPE,
HUGO MÜLLER,
FRANCIS R. JAPP,
RAPHAEL MELDOLA.

ON THE DISSOCIATION SPECTRA OF MELTED
SALTS.SULPHUR, PHOSPHORUS, AND THE SOLID
PHOSPHOROUS COMPOUNDS.

By A. DE GRAMONT.

Sulphur.

SALTS containing sulphur, sulphides or sulphates, when submitted to the action of the condenser spark in the apparatus I have previously described, give a very brilliant spectrum of the sulphur lines, the same as with solid metallic sulphides (mineral conductors, for instance), and free sulphur, either melted and solidified on two electrodes in the air, or contained in a Salet tube. The melted salts, however, have this advantage over minerals, that they can be freed from foreign matters which would complicate the spectrum, especially in the violet (principally iron and calcium lines). For the purpose of this investigation, I chose the alkaline sulphates in preference to the sulphides; the latter attacking to a certain extent the platinum wires and spatulas on which they are melted. If, in the dissociation spectra obtained, lines of considerable brilliancy are always present, the appearance of faint and hardly visible lines would seem to depend on the quality of the spark or on the resistance to dissociation of the compound under examination; and if, by corresponding variations of these two factors, we are enabled to obtain a complete line spectrum, we must not, on the other hand, be astonished at the absence of some of the fainter lines

at any particular moment, under known conditions. I will now give a complete list of the lines of sulphur compounds produced by the condenser discharge either with free sulphur, melted salts, or with minerals. The measurements, made with a spectroscopic with two prisms, have been corrected to the normal spectrum of Rowland. The Greek letters denoting the principal group are those used by Salet; I have only added ϕ and ψ for two groups of red lines easily seen in all sulphur compounds.

	657.6	Fairly well seen.
	645.4	Faint.
S ϕ	641.65	
	640.25	Well marked.
	639.0	
S ψ	632.0	Fairly strong.
	630.95	"
	629.1	"
S α	615.35	Faint; doubtful.
	601.65	Very faint; doubtful.
	586.7	Fairly well seen.
	581.75	Very faint.
	567.3	Fairly strong.
	566.3	Strong.
	565.2	"
	564.3	"
	562.3	Fairly well seen.
	560.85	Strong.
	558.4	Faint.
	556.95	"
S β	555.95	"
	552.85	"
	551.45	"
	550.85	Well marked.
S γ	547.35	Fairly strong.
	545.3	Strong.
	543.5	"
	543.0	Fairly strong.
S δ	534.35	Strong.
	532.1	"
	522.8	Well seen, but doubtful.
	521.95	Fairly strong.
	521.3	Strong.
	520.2	"
	516.1	Fairly well seen.
	514.35	Well marked.
	512.85	Faint.
	511.4	"
	510.3	Well marked.
	504.6	Well seen.
S ϵ	503.7	"
	503.25	Strong.
	502.2	Fairly strong.
	501.45	Well seen; diffuse.
S ζ	499.55	Fairly strong.
	494.2	Faint.
	492.55	Fairly strong.
	490.5	Fairly well seen; diffuse.
S η	488.5	Faint; vague.
	482.7	Hardly visible.
	481.5	Fairly strong.
	480.5	Faint.
S θ	479.4	"
	478.2	"
	476.6	Hardly visible.
	475.7	"
S μ	471.65	Fairly strong.
	466.6	Hardly visible.
	465.5	"
	463.3	Fairly well seen.
S ν	461.6	Faint; doubtful.
	456.2	Faint; diffuse.
	455.2	Strong.
	452.6	"
	448.5	"
	446.45	"

	442.85	Faint; diffuse.
	438.8	" "
	435.0	" "
	434.2	Hardly visible; doubtful.
	433.6	Faint; doubtful.
	432.8	" "
S π	429.55	Strong.
	428.4	" "
	427.9	Faint.
	426.95	Fairly strong.
	425.95	Faint.
	425.15	Fairly strong.
	424.15	Faint.
	422.55	" "
S ρ	417 (about)	Faint; diffuse.
	416	" "
	415	" "

I would here point out that I am not certain as to the sulphur lines that I have described as *doubtful*.

S ψ , formed of bright lines, is easily seen in all compounds of sulphur. S α , of which the strong lines are very close to the Group β of air, is very characteristic, but difficult to determine when arsenic and lead are also present, which frequently happens in the case of minerals. S β , S γ , S δ are the brightest and most characteristic groups in this spectrum, and are very distinct even with only one prism; the intercalation of the metallic lines hardly weakens them, with the exception of those of copper and lead in the case of S δ . But S γ appears to be the most persistent and most sensitive group of the spectrum. In spite of the brightness of its lines, S ϵ is not of much use in analysis, for it is mixed with the important Group α of air, from which it is very difficult to free it, even when the spark occurs in the body of the melted salt in the flame itself. The three blue lines S ζ , S η , S θ , relatively less strong in melted salts than in free sulphur, are, however, easy to recognise. The groups S μ and S ν are, on the contrary, often difficult to distinguish in sulphur compounds, where the condensation necessary to dissociate these latter blurs them so much that they can hardly be distinguished. I have, in the table, given the intensity of these lines in free sulphur. S ρ is only visible with a good deal of light and a non-absorbent apparatus. I have not been absolutely certain of it in any compounds.

To sum up, the groups which should be used for the identification of sulphur are:—Firstly, S β , S γ , S δ , in the green; and, secondly, S ϕ , in the red; S α , S ϵ , in the green, and S ζ , S η , S θ in the blue. I would, finally, remark that the lines have appeared to me to be more sharp, distinct, and less enlarged when using sulphates than sulphides. This difference, however, has not seemed to me to be sufficiently decisive to be perfectly characteristic.

Phosphorus.

The spectrum of phosphorus is one of the easiest to obtain by the dissociation of melted salts; the condensation obtained by one Leyden jar being sufficient to show it. The line spectrum thus produced is far better, both in sharpness and intensity, than that obtained by the use of Plücker or Salet tubes, which have, as in the case of sulphur, been exclusively used up to the present.

In this work I have principally used alkaline phosphates, and I now give the measurements of the wave-lengths of the phosphorus lines corrected to the normal spectrum of Rowland. The alphabetical descriptions of the lines are those used by Salet in his "Traité de Spectroscopie." The lines marked * were not observed by him.

	650.7	Easily seen; diffuse.
	645.9	Well marked.
	608.9	Easily seen.
P α	604.3	Very strong; bright.
	603.55	Fairly strong; bright.
	602.6	Very strong; bright.

P β	549.95	Fairly well seen.
	546.3	Faint.
	545.4	" "
P γ	542.45	Very strong.
	* 541.0	Strong.
	* 538.6	" "
	* 534.1	Strong; rather diffuse.
	531.2	" "
	529.3	" "
P δ	525.1	Very strong.
	* 496.9	Fairly well marked; diffuse.
	494.2	Well marked.
P ϵ	* 460.4	Very strong.
	458.95	" "

The difference in intensity between the lines marked *very strong* and those marked *strong* is not very great. The most characteristic and sensitive groups are:—First, the triple red P α and the double blue P ϵ , both of which are easily recognised at first sight; and, secondly, the series of green lines from P γ to P δ . Those which were not recorded by M. Salet correspond in intensity, and approximately in position, with the lines given by Plücker in the plate attached to his memoir, and from which Mr. Watts has deduced (approximately) their wave-lengths.

The Solid Phosphorous Compounds.

The solid phosphorous compounds, when subjected to the condenser spark, give at once in the cold a line spectrum of phosphorus as beautiful as that obtained from melted phosphates. A slight condensation is sufficient even for bodies which are non-conducting but volatilisable in the spark. The proportions between the relative intensities of the lines are the same as with the melted salts, which, as above, will furnish comparative spectra. By this means we can easily detect the phosphorus lines in metallurgical products, even when they are mixed with the metallic lines.

The phosphides of copper, for example, give a particularly brilliant spectrum of phosphorus, because of the small number of metallic lines present. Among the different phosphorous alloys, the large number of iron lines would appear at first sight to make the detection of phosphorus a difficult matter. It is, however, easily recognised by the characteristic triple orange line P α (604.3, 603.5, 602.6), whose brightness is almost equal to that of the iron lines, which are notably weak in this region. This reaction takes place perfectly plainly in alloys containing only 2 per cent of phosphorus, but it is not very distinct in the case of alloys containing smaller quantities; it becomes less and less certain until, at about one-thousandth, the lines are practically invisible. In the same manner, a metallic phosphide, insoluble in acids, isolated by M. Friedel from the Cañon el Diablo meteorite, gave me the principal phosphorus lines in the midst of those of iron and nickel.

Sulphophosphides or Thiophosphates.—As an example of the spectral examination of non-conducting solid bodies—which are, however, volatilisable in the electric spark—I will take the new series of sulphophosphides discovered by M. Friedel (*Comptes Rendus*, vol. cxix., 1894), and obtained by him in the crystalline form in sealed tubes at a high temperature; they correspond to the formulae $P_2S_6M_4$ or $P_2S_6M_{12}$. Through the kindness of M. Friedel, I was enabled to observe that their direct spark spectra are the same as those of the corresponding natural minerals, metallic sulphides, already described, but that they further show the phosphorus lines much more strongly and much brighter than those of sulphur, in spite of the fact that the percentage proportion present of this body is about three times as great as that of the phosphorus in this series of sulphophosphides. A condenser surface equal to a small Leyden jar of 5 or 6 square decimetres is sufficient to cause the spectrum of phosphorus to appear most distinctly, while it is necessary to use two or three jars (about 25 to 35 square decimetres) to obtain

that of sulphur—the intensity of which, under these conditions, never appears to be so great as with the corresponding minerals. Thus, with $P_2S_6Fe_2$ the sulphur lines are more brilliant and more easily seen than in pyrites; while with $P_2S_6Cu_4$ they are less bright than in chalcosine. $P_2S_6Ag_4$ is comparable with argyrose; $P_2S_6Pb_2$, with galena; and $P_2S_6Hg_2$, with cinnabar. All three of these are better conductors than the compounds of tin, $P_2S_6Sn_2$ and P_2S_6Sn , which require three jars to give a good spectrum of sulphur, and are by no means comparable with any corresponding mineral.

These examples will suffice to show the ease and simplicity of the direct spectral analysis of solid compounds, even when bad conductors. A little practice will enable one, by making slight variations in the arrangement of the apparatus, to apply this method to widely different substances.—*Bull. Soc. Chim.*, Series 3, vol. xix.-xx., No. 2.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING JANUARY 31ST, 1898.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, February 10th, 1898.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 182 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Jan. 1st to Jan. 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 182 samples examined by us, all were found to be clear, bright, and well filtered.

There has been a serious deficiency in the rainfall during the month of January, rain having fallen on five days only, viz., the 4th, 5th, 6th, 16th, and 31st, and then to only a small extent; the actual figures are:—Rainfall at Oxford, 0.82 inches; thirty years' average, 2.16 inches; deficiency, 1.34 inches.

Our bacteriological examinations of 251 samples have given the results recorded in the following table; we have also examined 40 other samples, from special wells, stand-pipes, &c., making a total of 291 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 26 samples) ..	2377
New River, filtered (mean of 26 samples) ..	19
Thames, unfiltered (mean of 26 samples) ..	4263
Thames water, from the clear water wells of five Thames-derived supplies (mean of 123 samples)	52
Ditto ditto highest	386
Ditto ditto lowest	0
River Lea, unfiltered (mean of 26 samples) ..	2866
River Lea, from the East London Company's clear water well (mean of 24 samples) . .	25

For the time of year, the rivers Thames and Lea are in exceptionally good condition, owing to the lack of rain, but at the same time it is satisfactory to see from the above results that the filtering plant of the various companies is in good working order, and that with the exception of one or two abnormally high results, which after all did not include pathogenic organisms, the bacterial quality of the water supplied to London is excellent.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

SALTS OF DINITRO-ALPHA-NAPHTHOL WITH VARIOUS METALLIC BASES.*

By T. H. NORTON and H. LOWENSTEIN.

THE preparation and study of the following compounds were undertaken with the view of ascertaining whether in addition to the few salts of dinitro- α -naphthol thus far known, there might not be other combinations with metallic bases possessing a greater degree of solubility than the commercial calcium salt (Manchester yellow, Martius Gelb) now so largely used, or more especially showing distinct variations in the colour imparted to animal and vegetable fibres. The results showed that in the latter respect the metal present is practically without influence on the tinctorial properties of dinitro- α -naphthol, the tint yielded in each individual case being essentially that imparted by the free phenol or by its calcium salt.

The salts of dinitro- α -naphthol thus far examined and analysed are those with sodium, potassium (Hübner, *Ann. Chem.* (Liebig), ccviii., 332), ammonium, barium, strontium (Martius, *Journ. Prakt. Chem.*, cii., 443), calcium (Darmstädter and Wichelhaus, *Ann. Chem.* (Liebig), clvii., 201; Liebermann, *Ann. Chem.* (Liebig), clxxxiii., 249; Martius, *vide supra*), and silver (Hübner, *Ann. Chem.* (Liebig), ccviii., 332).

The dinitro- α -naphthol used was prepared by precipitating the concentrated aqueous solution of the commercial calcium salt by dilute sulphuric acid, washing the precipitate thoroughly with water, drying, and extracting the liberated phenol by boiling alcohol. The finely crystallised product thus obtained possessed the melting-point of 138°.

Lithium Dinitro- α -naphtholate, $C_{10}H_7(NO_2)_2OLi$.

This salt is obtained in solution by boiling an excess of dinitro- α -naphthol with an aqueous solution of lithium carbonate and filtering. On evaporation it is deposited in the form of a brilliant crimson powder, which is amorphous and anhydrous. The salt, on being heated to 295°, explodes before melting. It is soluble in 96 parts of water at 19°, in 14 parts of boiling water, in 50 parts of cold alcohol, in 15 parts of boiling alcohol, and in 300 parts of ether. It is insoluble in carbon disulphide and in benzene. The temperature of solutions, unless otherwise noted, is 18° C.

The salt, dried between sheets of bibulous paper, lost no weight on heating to 105°. The analyses were made by adding concentrated sulphuric acid to weighed amounts in a platinum crucible, evaporating to dryness, and ignition as lithium sulphate. Care is necessary to avoid spattering.

I. 0.246 grm. of the dry salt gave 0.0535 grm. lithium sulphate, equal to 0.0069 grm. of lithium, or 2.80 per cent.

II. 0.256 grm. gave 0.0535 grm. lithium sulphate, equal to 0.0068 grm. of lithium, or 2.78 per cent.

* Contributions from the Chemical Laboratory of the University of Cincinnati. From the *Journal of the American Chemical Society*, xix., No. 12.

	Calculated for $C_{10}H_7N_2O_5Li$.	I.	Found.	II.
Lithium ..	2.87	2.80	2.88	

Magnesium Dinitro- α -naphtholate, $[C_{10}H_5(NO_2)_2O]_2Mg$.

This salt is prepared by boiling in water a mixture of magnesium carbonate with an excess of the naphthol. It crystallises slowly from the filtered solution in the form of reddish needles, grouped in rosettes. It is anhydrous, the salt dried between sheets of bibulous paper, losing no weight when heated to 105°. It is soluble in 792 parts of water at 20°, in 154 parts of boiling water, in 436 parts of cold alcohol, in 144 parts of boiling alcohol, and in 260 parts of ether. It is insoluble in benzene and carbon disulphide.

The first three analyses were made by igniting the substance in a platinum crucible, in which a smaller inverted crucible was wedged, so as to prevent as much as possible mechanical loss by explosive decomposition, and weighed as magnesia.

In the fourth analysis the substance was strongly heated in a sealed tube. After cooling, the tube was opened, its contents were dissolved in dilute nitric acid, and the solution was evaporated to dryness, ignited, and weighed likewise as magnesia.

- I. 0.035 grm. of the salt dried at 105° gave 0.0025 grm. magnesium oxide, equal to 0.0015 grm. magnesium, or 4.28 per cent.
- II. 0.1557 grm. gave 0.0113 grm. magnesium oxide, equal to 0.0067 grm. magnesium, or 4.35 per cent.
- III. 0.1552 grm. gave 0.012 grm. magnesium oxide, equal to 0.0072 grm. magnesium, or 4.64 per cent.
- IV. 0.0623 grm. gave 0.0039 grm. magnesium oxide, equal to 0.0023 grm. magnesium, or 3.77 per cent.

	Calculated for $(C_{10}H_7N_2O_5)_2Mg$.	I.	Found.	III.	IV.
Magnesium ..	4.87	4.28	4.35	4.64	4.77

Zinc Dinitro- α -naphtholate, $[C_{10}H_5(NO_2)_2O]_2Zn$.

This is obtained by boiling together in water an excess of the naphthol with zinc carbonate. From a hot concentrated solution it crystallises out in the form of handsome reddish yellow needles, while more dilute solutions yield reddish crystals, possessing the shape of rhomboidal plates. In this respect the zinc salt resembles the calcium salt (Liebermann, *Ann. Chem.* (Liebig), clxxxiii., 249).

It is soluble in 960 parts of water at 20°, in 300 parts of boiling water, in 250 parts of cold alcohol, in 115 parts of boiling alcohol, and in 150 parts of ether. It is insoluble in benzene and carbon disulphide. The salt is anhydrous; crystals dried between sheets of bibulous paper losing no weight at 105°.

The explosive nature of the compound renders necessary in analysis the careful use of the two crucibles described above, small amounts being taken. Even with these precautions a small loss is unavoidable.

- I. 0.123 grm. of the salt dried at 105° gave 0.017 grm. zinc oxide, equal to 0.0136 grm. zinc, or 11.09 per cent.
- II. 0.108 grm. gave 0.016 grm. zinc oxide, equal to 0.0128 grm. zinc, or 11.89 per cent.

	Calculated for $(C_{10}H_7N_2O_5)_2Zn$.	I.	Found.	II.
Zinc ..	12.22	11.09	11.89	

Copper Dinitro- α -naphtholate, $[C_{10}H_5(NO_2)_2O]Cu$.

This salt is obtained by adding an excess of a concentrated solution of copper chloride to a cold aqueous solution of ammonium dinitro- α -naphthol. The chocolate coloured precipitate is washed with water until all traces of copper chloride are removed, and dried between sheets of bibulous paper. Thus prepared, it is an anhydrous dark brown

amorphous powder, the most insoluble of the salts of the naphthol thus far known.

It dissolves in 5750 parts of water at 21°, in 2165 parts of boiling-water, in 1007 parts of cold alcohol, in 954 parts of boiling alcohol, and in 2615 parts of ether. It is insoluble in benzene and carbon disulphide.

The analyses were performed by gently heating the salt with concentrated nitric acid in a covered porcelain crucible, and finally igniting. As in the preceding cases, the explosive nature of the salt renders the utmost care necessary.

- I. 0.2663 grm. of the salt dried at 105° gave 0.036 grm. cupric oxide, equal to 0.0287 grm. copper, or 10.79 per cent.
- II. 0.1665 grm. gave 0.023 grm. cupric oxide, equal to 0.0183 grm. copper, or 11.03 per cent.
- III. 0.3033 grm. gave 0.0443 grm. cupric oxide, equal to 0.0353 grm. copper, or 11.67 per cent.

	Calculated for $(C_{10}H_7N_2O_5)_2Cu$.	I.	Found.	III.
Copper ..	11.97	10.79	11.03	11.67

Solubilities of the Ammonium and Calcium Salts.

No data existing on the solubilities of these two compounds, the following determinations were made:—

Ammonium dinitro- α -naphtholate is soluble in 600 parts of water at 25°, in 38 parts of boiling water, in 113 parts of cold alcohol, in 57 parts of boiling alcohol, and in 1124 parts of ether.

Anhydrous calcium dinitro- α -naphtholate is soluble in 1656 parts of water at 20°, in 86 parts of boiling water, in 47 parts of cold alcohol, in 15 parts of hot alcohol, and in 45 parts of ether.

THE RELATION OF THE TASTE OF ACIDS TO THEIR DEGREE OF DISSOCIATION.

By THEODORE WILLIAM RICHARDS.

SINCE no systematic attempt appears to have been made to determine acids quantitatively by means of the sense of taste alone, it seemed worth while to carry out the following series of experiments with this idea in view.† The peculiar sourness which is distinctive of acids is probably to be referred to the hydrogen ion, which also is distinctive of them, and it becomes a question of no small interest to determine how closely the taste corresponds to the degree of dissociation.

The sense in question is well known to be rather an uncertain one, depending upon the habits and physical condition of its possessor, so that an experimenter could hardly expect to obtain absolute results from its use. On the other hand, relative taste, determined by alternately tasting two solutions, might well furnish some clue as to the relative strength of these solutions.

The water used in the experiments recorded below was distilled in a platinum still over alkaline permanganate, the first portions of the distillate being rejected. For the final experiments this water was re-distilled; in this way an unusually tasteless liquid may be obtained. The solutions of acids, alkalies, and salts were carefully made of pure materials. The test was accomplished by simply taking a small mouthful (5 c.c.) of the liquid in question (which is advantageously warmed beforehand to 40°), and allowing this liquid to remain upon the tongue for a few seconds until a constant intensity of taste is secured. A

* Contributions from the Chemical Laboratory of Harvard College. From the *American Chemical Journal*, vol. xx., No. 2.

† Dr. Squibb has used this physiological method upon cocaine with good results. ("Ephemeris of Materia Medica, &c.," Brooklyn, N.Y., vol. iii., No. 1, p. 918, 1887). My attention was called to this interesting work after the present paper was written, and I know of no similar investigation.

few of the individual experiments are recorded below in order to define the method, but most of them are merely indicated in the conclusions.

(1) To four several portions of water, each measuring 10 c.c., were added respectively 0.05, 0.10, 0.20, and 0.30 c.c. of decinormal hydrochloric acid. The first of these solutions remained unchanged in taste, the second became faintly acid, and the others very distinctly so.

(2) In order to test the degree of accuracy attainable in this way, a number of beakers containing dilute acid were "shuffled," so that the identity of each was lost, and the attempt was then made to arrange them according to strength by means of the taste alone. Small inconspicuous labels showed finally how successful the attempt had been. In the first trial, four beakers, each containing 30 c.c. of water, were acidified by adding 0.2, 0.4, 0.6, and 0.8 c.c. of decinormal hydrochloric acid. These were arranged by taste without great deliberation in their proper order, showing that the method is capable of an accuracy of 25 per cent, even with a comparatively unpractised tongue.

(3) Another similar trial was made with similar amounts of water containing 0.3, 0.4, 0.5, and 0.6 c.c. of decinormal acid respectively. After repeated tasting and much deliberation these were arranged in the order 1, 2, 4, 3; thus my success with the method did not go beyond 20 per cent, although the limit might unquestionably be reduced by practice.

Similar experiments with sulphuric, nitric, and hydrobromic acids showed that the sensitiveness in these cases is about the same—an acid of the concentration of somewhat less than $\frac{1}{100}$ normal, representing the limit of possible detection as indicated by my sense of taste. This is a very weak solution, containing less than a milligram of hydrogen ions in a litre.

It is, of course, possible that a part of this taste might be due to the negative ion. Further light upon this point is obviously to be obtained by neutralising the acid with potash or soda. It was found in this way that an acid liquid which tasted distinctly sour became absolutely tasteless upon neutralisation; indeed, a centinormal solution of potassium chloride has less taste than a millinormal solution of acid. According to the dissociation hypothesis, the hydrogen ions have been removed by the neutralisation, and we are now tasting potassium and chlorine ions, which are either not very strong in taste, or else have the peculiar property of nearly eliminating one another's taste by mixture. Since most properties of solutions are additive, and we cannot well conceive of a minus taste (except such a subtraction from the taste as might be caused by a partial paralysis of the nerves, owing to some very powerful excitement), the former alternative seems to be the most natural one; hence we may believe that neither potassium nor chlorine ions possess as strong a taste as those of hydrogen.

It becomes now a matter of interest to compare these results with those given by very dilute solutions of less ionised acids. Tartaric acid thus diluted, tasted somewhat less sour than a corresponding solution of the mineral acids, citric acid was still less sour, and acetic acid was the least sour of all. While qualitatively these observations agree with those which we should expect to make, the quantitative agreement is less satisfactory. Acetic acid, for instance, seemed to taste at least as strong as hydrochloric acid of one-third its concentration, whereas at a concentration of $\frac{1}{100}$ normal, Kohlrausch's experiments upon its electrolytic conductivity show that only about one-fourteenth of the acid is dissociated.* The reason for this discrepancy is not evident, unless the act of tasting removes the dissociated part of the acid, and so leaves room for further dissociation, or unless the undissociated acid possesses a taste resembling that of the

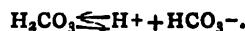
hydrogen ions, but weaker. Both of these hypotheses carry one too far into physiological chemistry to receive treatment here.

Somewhat more satisfactory were the results of mass action upon the taste. The addition of small amounts of potassic chloride to very dilute solutions of hydrochloric acid produced no appreciable effect upon the taste, while the sourness of acetic acid was noticeably diminished, and that of hydrochloric acid was almost destroyed by adding similar amounts of potassic or sodic acetates. These phenomena are qualitatively in accord with the dissociation hypothesis and the mass law, which tell us that in such a reaction as



the addition of a large proportion of acetic ions from the almost wholly dissociated sodic acetate is capable of forcing the reaction backwards and destroying the hydrogen ions. The case is, of course, analogous to the effect of salts in modifying the catalytic action of acids upon cane-sugar and esters, as measured by Ostwald (*Journ. Prakt. Chem.*, 1883, 28, 449; also 1885, 31, 307). Arrhenius (*Zeit. Physikal. Chem.*, 4, 226), Spohr (*Journ. Prakt. Chem.*, 1885, 32, 32), Trevor (*Zeit. Physikal. Chem.*, 10, 321), Palmaer (*Ibid.*, 22, 492), and others. We should expect, however, to find the taste of the acetic acid wholly destroyed by the addition of sodic acetate, because the acid's power of inverting sugar is so much diminished thereby; and the fact that some acid taste remains is only to be accounted for by some such hypothesis as those given above. The fact that the taste of hydrochloric acid is diminished much more than that of acetic acid by the addition of sodic acetate is not inconsistent with these hypotheses; for acetic acid, whether wholly or partly undissociated, has been shown to be much less sour than hydrochloric acid; and acetic acid is undoubtedly formed in this case.

A common application of these principles is to be found in the preparation of the much-used "soda water" or "mineral water" of commerce. Unless a small amount of sodic carbonate is added during its preparation, the effervescent solution is so sour as to be unpleasant to some tastes. This sourness may sometimes have been augmented by a stronger acid carried over in the process of careless preparation; but the fact that it exists in some degree, even in carefully prepared carbonic acid water, was shown by experiment in the laboratory. In any case the sourness is removed by sodic bicarbonate, and the hypothetical explanation is similar to that given above. The following equilibrium is usually assumed to exist in the solution of carbonic acid:—



The acid is, however, only very slightly dissociated. Hydric sodic carbonate, on the other hand, is probably largely dissociated into Na^+ and HCO_3^- , and when this salt is added these latter ions force back the already very slight dissociation of the carbonic acid, rendering the solution pleasant and soft to the taste by removing some of the hydrogen ions.

Since sodic chloride was found to possess a taste so much feebler than that of hydrochloric acid, it is evident that the sense of taste might form a fairly sensitive indicator of the end-point of an acidimetric reaction. It is possible easily to detect sourness in a millinormal solution of acid, hence in working with decinormal solutions of acids and alkalis we ought to be able to determine the end-point to within 1 or 2 per cent.

Accordingly several titrations were made with exactly decinormal solutions of hydrochloric acid and sodic hydroxide, the liquid being tasted with a glass rod until the end-point was nearly reached, when small mouthfuls were tasted. The alkalinity of saliva is insufficient to make any essential difference in the results. If large amounts of liquid are removed, it is obviously necessary to allow for the acid or alkali thus taken out; but this

* At 18°, when $\kappa = 167 \mu = 17.0$
 $\kappa = 500 \mu = 25.3$
 $\kappa = \infty \mu = 318.4$ } *Wied. Ann.*, 26, 161, 1885.

correction may be reduced to a very small amount by the use of large quantities of the original solutions.

The best results were obtained by adding slightly too much acid, and then adding alkali until the sour taste just disappeared. The figures are given below—the first and second columns containing the number of cubic centimetres of acid and alkali respectively, and the third the percentage of acid found.

Acidimetric Titration.

C.c. of acid taken.	C.c. of alkali taken.	Per cent of acid found.
7'00	6'99	99'9
10'73	10'67	99'4
7'21	7'15	99'1
14'96	14'99	100'2
14'44	14'59	100'9
	Average	99'9

The accuracy of these results, which were obtained in an unprejudiced fashion, is somewhat astounding. Evidently, in the course of the few experiments described here, the sense of taste had been noticeably sharpened. To what limit this sensitiveness might be carried it is hard to predict; but already the method of analysis had assumed a degree of accuracy exceeding some which are seriously applied as quantitatively useful.

Efforts were made also to analyse the taste produced by weak currents of electricity, as well as the effect of sugars in mitigating the unpleasantness of great acidity of taste, but the complex physiological relations involved in these and similar cases led to an unsatisfactory outcome.

The results of this paper, although they are only qualitatively consistent with the theory of dissociation, nevertheless show that the sense of taste might be used more precisely in the laboratory than is usually believed.

THE ELECTROLYTIC DETERMINATION OF CADMIUM.*

By DANIEL L. WALLACE and EDGAR F. SMITH.

ABOUT a year ago Max Heidenreich (*Ber. d. Chem. Ges.*, xxix., 1585) published an article in which he reviewed methods proposed by one of us (S.) for the electrolytic determination of certain metals. In some instances he confirmed the observations of Smith, but in most cases differed with him. Recently S. Avery and Benton Dales (*Journ. Amer. Chem. Soc.*, xix., 380) announced that so far as the determination of cadmium was concerned their "result was in complete harmony with the experiments of Heidenreich." In consequence of these experiences with the electrolytic methods suggested by Smith for the determination of cadmium, we have repeated them, and offer new results obtained by us.

It was in 1878 (*Proc. Am. Phil. Soc.*, Nov., 1878) that Smith stated that "0'1450 gm. of cadmium oxide was dissolved in acetic acid, the excess of the latter expelled upon a water-bath, and the platinum crucible then about half filled with water, and . . . connected with the negative pole of a two-cell Bunsen battery. . . . The deposition of the cadmium was regular . . . a perfectly crystalline, greyish white layer." The precipitation was complete in about three hours. The experiment was repeated, but a bichromate battery was substituted for the Bunsen cells. At the time when these trials were made, current strength in definite units was not given: hence subsequent workers have met with difficulty in repeating some of the early work, and perhaps that may account for the failure to precipitate cadmium from the solution of its acetate. We have sought to obtain the original

working conditions given by Smith, have stated them in definite units, and append results obtained by their observance.

Experiment 1.—0'1329 gm. of cadmium oxide was dissolved in acetic acid, the solution was evaporated to dryness, and the residue dissolved in 30 c.c. of water. The liquid was then heated to 50° C., and electrolysed with a current of 0'02 ampère for 37 sq. c.m. of cathode surface. Voltage 3'5. The metal was completely precipitated in four hours. It was crystalline and perfectly adherent. There was no evidence of sponginess. The hot acid liquid was syphoned off, and replaced by water without interrupting the current. We found that the period of precipitation could be diminished, and very good results be obtained by adding 1 gm. of ammonium acetate to the solution when the current had acted for an hour. The quantity of metallic cadmium present in this particular experiment was 0'1162 gm., while that actually found equalled 0'1158 gm.

Experiment 2.—0'1332 gm. of oxide, treated precisely as outlined in Experiment 1, gave 0'1164 gm. of adherent crystalline metal instead of 0'1165 gm.

With these results before us we see no cause why the deposition of this metal from its acetate solution should be condemned. Neumann (*"Theorie und Praxis der Analyt. Elektrolyse,"* p. 129) mentions it as being possible, and Yver (*Bull. Soc. Chim.*, xxxiv., 18) even used an acetate solution in the electrolytic separation of cadmium from zinc.

Smith (*Am. Chem. J.*, ii., 41) published experiments made by him on the precipitation of cadmium from solutions containing free sulphuric acid. As this method seems not to have proved satisfactory in the hands of Heidenreich, and of Avery and Dales, we have repeated the work, and offer the following experiments as evidence in favour of the Smith method.

Experiment 1.—0'1270 gm. of cadmium oxide was dissolved in 2 c.c. of sulphuric acid, sp. gr. 1'09, and the solution then made up with water to 30 c.c. It was heated to 50° C., and electrolysed for a period of 4½ hours with a current of 0'08 ampère for 37 sq. c.m. cathode surface. Voltage 2'5. The deposit weighed 0'1105 gm. instead of 0'1111 gm. It was crystalline and adherent. Tendency to sponginess was not observed. The acid liquid was syphoned off before interrupting the current. The filtrate showed no signs of cadmium when tests were made for it.

Experiment 2.—In this trial 0'1358 gm. of cadmium oxide was dissolved and treated as in Experiment 1. The deposit of metal weighed 0'1181 gm. instead of 0'1188 gm.

We have never experienced any difficulty in the application of this method, and are also pleased to observe that Neumann (*loc. cit.*) gives an example confirming the early recommendation of Smith. It is this: "die Lösung von 0'3 gm. Kadmium sulfat in 150 c.c. Wasser mit 1 bis 2 c.c. verdünnter Schwefelsäure versetzt, wird auf 70 bis 80 Grad C., erwärmt und bei diesem Temperatur durch Ströme von 0'6 bis 1 ampère zersetzt. Die Spannung schwankt . . . zwischen 2'5 bis 5 volt . . . Der Niederschlag ist silberweiss."

Our experiences with the precipitation of cadmium from a phosphoric acid solution will be reserved for a subsequent communication.

We regard the double cyanide solution (*Ber. d. Chem. Ges.*, xxv., 779) as the best adapted for the deposition of cadmium. Further evidences of this are the many atomic weight determinations made of this metal, in which this particular solution was employed (*Ztschr. Anorg. Chem.*, i., 364; *Journ. Amer. Chem. Soc.*, xviii., 1022).

The first suggestion for the electrolytic separation of copper from cadmium was also made by Smith (*Am. Chem. J.*, ii., 43). He demonstrated that, in the presence of free nitric acid, copper was deposited in satisfactory form, while the cadmium continued in solution,

* Contribution from the John Harrison Laboratory of Chemistry. From the *Journal of the American Chemical Society*, xix., p. 870.

Heidenreich (*loc. cit.*), in speaking of this method, which has received frequent confirmation, remarks: "eine Reihe von Versuchen, Kupfer von Cadmium aus einer freien Salpetersäure enthaltenden Lösung zu trennen, führten zu keinem befriedigenden Ergebnisse." In reply to this we offer the following experiments:—

Experiment 1.—To a solution containing 0.3893 grm. of copper sulphate (= 0.0988 grm. of copper) and 0.1152 grm. of cadmium oxide (= 0.0985 grm. of cadmium) were added 2 c.c. of nitric acid, sp. gr. 1.43. The total dilution of the solution equalled 100 c.c. It was heated to 50° C., and electrolysed with a current N.D.₁₀₀ = 0.10 ampère. Voltage 2.5. The metal was completely precipitated in three hours. It was bright in colour, and satisfactory in every respect. It weighed 0.0988 grm. and did not contain cadmium.

Experiment 2.—A solution containing the same quantity of copper and 0.1203 grm. of cadmium oxide, when treated exactly as in Experiment 1, gave 0.0987 grm. of copper. It was free from cadmium.

To these results we would add the observation of Neumann ("Electrolyse," p. 169): "Am einfachsten ist zur Trennung von Kupfer und Cadmium immer die Verbindung der mit freier Salpetersäure versetzten Lösung beider Salze."

Heidenreich maintains that the separation of copper from cadmium succeeds "sicherer aus einer mit Schwefelsäure versetzten Lösung." This solution had been previously recommended by Smith (see "Electrochemical Analysis," 2d ed., p. 108), and his observations were confirmed by Freudenberg (*Ztschr. Phys. Chem.*, xii., 97).

The results recorded in the preceding paragraphs justify us in saying that cadmium can be successfully determined electrolytically when working either with its acetate or sulphate, and that its separation from copper in the presence of nitric acid is in every respect satisfactory.

The failure of Heidenreich to precipitate uranic oxide by means of the current will be considered later.

TESTING OF FORMALDEHYD.*

By CARL E. SMITH.

THE rapidly increasing uses of formaldehyd make it desirable that standards be established for the strength and purity of the commercial products. A method of assay, simple and rapid, as well as reasonably accurate, is needed as a guide for manufacturer and pharmacist, as also tests for the various impurities liable to be present, and reactions to establish its identity. As the commercial solutions vary considerably in quality, ready means should be at hand for controlling the quality of the pharmacist's supply.

Assay Methods.

A method of assay, to be generally applicable, must not be affected in accuracy by the presence of ordinary impurities frequently contained in commercial formaldehyd solution, such as methyl alcohol and acetone; neither should it require much time and attention or complicated operations. Of the number of methods which have been published within recent years, the principal ones were examined, in order to ascertain which of them approaches nearest to these requirements, with the results stated below:—

Hydroxylamine Method.—Proposed by Brochet and Cambier (*Compt. Rend.*, vol. 120, p. 449, and *Ztsch. f. Anal. Chem.*, vol. 34, p. 623). Based on the reaction of hydroxylamine hydrochloride and formaldehyd, with the

liberation of hydrochloric acid, according to the following equation:—



The formaldehyd entered into combination is determined by the amount of acid set free.

In the experiments made the details prescribed by the authors were followed, except that N/10 soda solution was used for titrating the hydrochloric acid instead of N/10 borax solution.

Ten c.c. of a solution containing 0.0864 grm. of a concentrated formaldehyd solution were mixed in a small flask, with 10 c.c. of a 2.5 per cent solution of hydroxylamine hydrochloride. (The hydroxylamine salt is about five times the amount involved in the reaction. This large excess was afterwards found unnecessary, 50 per cent excess being quite sufficient). After standing ten minutes the liberated acid was titrated, requiring, after deducting 0.1 c.c. for free acid in the hydroxylamine solution, 10.7 c.c. of N/10 soda; 1 c.c. = 0.003 grm. formaldehyd.

$$\frac{10.7 \times 0.003 \times 100}{0.0864} = 37.2 \text{ per cent.}$$

Several trials were made to determine the time limit of the reaction.

Allowing twenty minutes to complete the reaction, the result was 37.2 per cent.

Allowing forty-five minutes to complete the reaction, the result was 37.3 per cent.

Other trials showed that shaking the mixture does not materially hasten the reaction, and that it is practically complete in seven to eight minutes after admixture.

The above figures agree closely with those obtained on the same solution by other methods.

The method is quick and accurate, when applied to pure solutions, but the accuracy is interfered with by the presence of other aldehyds and acetone, as stated by G. Romijn (*Ztsch. f. Anal. Chem.*, vol. 36, p. 18). For acetone the statement was verified by a simple test-tube experiment; aldehyds were not tried.

Iodine Method.—Proposed by Romijn (*ibid.*). Nearly identical with Messenger's process for estimating acetone, and therefore unsuitable for the assay of solutions liable to contain acetone. It was used only to determine the strength of pure solutions, made from para-formaldehyd, and for this purpose was found accurate and convenient. A very dilute solution of the formaldehyd is mixed with an excess of N/10 iodine solution, and caustic alkali solution added until the iodine is decolourised. After standing ten minutes dilute acid is added to liberate the excess of iodine, and this estimated with sodium hyposulphite.

Cyanide Method.—Also devised by Romijn (*ibid.*). It is based upon the formation of an addition product of formaldehyd and potassium cyanide, from which the cyanide cannot be precipitated with silver nitrate. It involves the use of standard solutions of silver nitrate, potassium cyanide, and potassium sulphocyanate. This method requires much more care and attention than any of the others tried, and a few trials showed conclusively that it is not likely to give satisfactory results except in practiced hands. It was, therefore, considered useless to proceed further with it.

Fixed-Alkali Method.—This consists in heating the formaldehyd with sodium or potassium hydrate solution under pressure, in a manner similar to that in the saponification of esters. The formaldehyd is converted into methyl alcohol and formic acid as follows:—



The method was most satisfactory when conducted as follows:—Three grms. of the sample are placed into a strong bottle of 50 c.c. capacity, with 25 c.c. of N/1 soda solution, the bottle closed with a tight-fitting rubber stopper, this tied down with a cord, and the bottle, after

* Report of Research Committee DII., Committee of Revision of the United States Pharmacopoeia. From the *American Journal of Pharmacy*, lxx., No. 2.

wrapping with a cloth, immersed in boiling water for one-half hour. After cooling, the excess of soda is titrated with N/1 sulphuric acid and phenolphthalein, each c.c. of soda solution consumed indicating 0.06 gm., or, if 3 grms. be taken, 0.5 per cent of formaldehyd.

The time required to complete the reaction is very much less than is stated by others who have tried the method, nor were some other stated disadvantages noticed, such as thickening or resinifying of the solutions. The results were reasonably accurate, except in the case of one commercial sample. The following figures illustrate the duration and degree of heat necessary. They were all obtained with the same formaldehyd solution:—

Heated in boiling water.	Per cent.	Heated over boiling water.	Per cent.
15 minutes ..	37.1	15 minutes ..	32.5
30 " ..	37.3	30 " ..	34.5
1 hour ..	37.6	1 hour ..	37.1
2 hours ..	37.2	2 hours ..	37.7
		3 " ..	37.3

On prolonged heating the solution frequently darkens so as to make dilution necessary before titrating.

The influence of the presence of acetone and methyl alcohol on the accuracy of the methods was determined.

(To be continued).

NOTICES OF BOOKS.

Bibliography of the Metals of the Platinum Group. Platinum, Palladium, Iridium, Rhodium, Osmium, Ruthenium, 1748—1896. By JAS. LEWIS HOWE. Smithsonian Miscellaneous Collections, No. 1084. City of Washington: published by the Smithsonian Institution. 1897. 318 pp., 8vo.

By the industry and ability of Professor Jas. Lewis Howe, M.D., Ph.D., of Washington, and Lee University, in Virginia, and through the liberality of the Smithsonian Institution, chemists are furnished with another invaluable work of reference, having the above title.

The well-printed volume contains about 2300 references to original papers, and probably nearly twice as many references to reproductions and abstracts of the same. These references are preceded by a list of the one hundred periodicals examined, and are followed by two exhaustive indices—a classified subject-index and an alphabetical author-index.

This bibliography forms one of a series compiled under the auspices of the Committee on Indexing Chemical Literature of the American Association for the Advancement of Science, and will prove indispensable to chemists working on the metals named. H. C. B.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

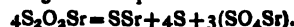
Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 5, January 31, 1898.

M. Cremona was elected a correspondent for the Geometrical Section, in succession to the late M. Brioschi.

Composition of Air in different Places, and the Density of the Gases.—A. Leduc.—The air in Paris contains 234 parts by weight of oxygen per 1000; in Havre, 230.5; at sea, 232.1; at Nice and Algiers, 232.3; while that in London contains only 231 parts. The densities of oxygen,

atmospheric nitrogen (which is a mixture), pure nitrogen, carbonic oxide, and carbonic acid are given, both in comparison with air and with oxygen. They agree very closely with Lord Rayleigh's results.

Decomposition of Hyposulphite and Sulphite of Strontium by Heat, and the Production of Phosphorescent Strontic Sulphide.—J. R. Mourello.—When hyposulphite of strontium is decomposed by heat a mixture of sulphate of strontium, sulphide of strontium, and sulphur is always formed, of which the relative quantities produced depend on the temperature reached and the time of the operation. When the heating is properly regulated, it is not difficult to obtain a complete decomposition according to the equation—



It possesses the maximum yellowish green phosphorescence when it contains 15.02 parts of sulphide of strontium, 16.12 parts of sulphur, and 68.45 parts of sulphate of strontium. The success of the operation depends on arresting the oxidation at a definite point. If sulphite of strontium is heated under the same conditions it is transformed entirely into sulphate of strontium; but, as before, by properly regulating the temperature, the phosphorescent mixture is obtained according to the formula $4\text{SO}_3\text{Sr} = 3(\text{SO}_4\text{Sr}) + \text{SSr}$, the best proportion is found to be 14.05 parts of sulphide of strontium and 85.94 parts of sulphate of strontium.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xvii.-xviii., No. 24.

M. Desgrez announced that, even in the cold, chloroform decomposes when in contact with a weak solution of potash, giving no longer formic acid, but carbonic oxide and water; chloral and bromoform give the same reaction; but iodoform, methyl-, and phenyl-chloroform do not give off any gas. The author feels sure that this formation of carbonic oxide in an aqueous alkaline solution could be profitably used in the toxicological search for chloroform.

M. Muttelet, by causing the para-nitrated derivative of chloride of benzoyle to react on the mono-substituted ortho-diamines and on the nitrated derivatives of the ortho-diamines, has obtained nitro-amidines, which are isomers of the nitro-amidines already described by him (*Bull. Soc. Chim.*, xvii., p. 865).

MM. Labbé and Flatau made a communication on some new crystalline compounds, by means of which they are enabled to distinguish, with certainty, geraniol and citronnellol.

While studying the different combinations which occur in steel on the addition of various elements, such as P, As, Si, Cr, Mn, M. Carnot has been able to isolate a certain number of definite compounds by attacking the steels, either with dilute acids or with chloride of copper.

M. Delépine has observed that when aldehyde of ammonia is dried *in vacuo* over sulphuric acid, it loses the elements of water and gives ethylidene-imine, hitherto unknown.

On the Preparation and Properties of the Borides of Calcium, Strontium, and Barium.—H. Moissan and P. William.—Already noticed in this column.

Application of the Friedel and Crafts Method to the Preparation of Acetones and Aromatic Aldehyds.—L. Bouveault.—A controversial paper concerning one recently published by M. Verley.

On the Nature of the Combination of Antipyrine with the Aldehyds.—G. Patein.—The author has endeavoured to establish, firstly, whether antipyrine really gives two kinds of compounds with formic aldehyd in particular and the aldehyds in general; and, secondly, if chloral is also capable of giving two kinds of compounds, and he finds that the aldehyds combine with antipyrine in the proportion of one molecule of aldehyd to two mole-

cules of antipyrine: this manner of combination is the only one, and the union is always made by means of the carbon, and never by the nitrogen. This does not apply to the chlorine derivatives, and chloral can only unite with antipyrine by means of the nitrogen.

The Action of Chloride of Benzoyl-*p*-nitratd on the Mono-substituted Ortho-diamines.—F. Muttelet. —The author describes the action of chloride of benzoyl-*p*-nitratd in excess, and hot, on the following ortho-diamines:—*o*-amido-phenyl-aniline, *o*-amido-phenyl-*p*-toluidine, *p*-nitro-*o*-amido-phenyl-aniline, and *p*-nitro-*o*-amido-phenyl-*p*-toluidine, and also the properties of each of these new nitro-amidines.

The Saccharines.—P. Monnet and J. Koetschet.—A reply to the paper recently published by M. Sisley (p. 84, 1897), in which he claims priority for the preparation of colouring matters derived from saccharine, described by the authors in the same publication (p. 690, 1897).

MISCELLANEOUS.

The Manchester Steam-Users' Association has recently issued a large printed sheet, suitable for display in boiler-houses, headed "Advice to Boiler Attendants," wherein will be found precise instructions as to the proper working of, and attending to steam-boilers, both with regard to water-level, lighting fires, smoke prevention, emptying boilers, overhauling, cleaning and inspecting, &c. Also a list of "Warnings" as to possible accidents and their causes; also what to do and what not to do in such cases: it is naively remarked that the boiler attendant may prefer to retire at such a time; he should certainly not expose himself unnecessarily in front of the furnaces, but should warn others of the danger.

Royal Institution.—On Thursday next (March 3rd) Professor J. A. Fleming, F.R.S., will deliver the first of a course of five lectures at the Royal Institution, on "Recent Researches in Magnetism and Diamagnetism"; and on Saturday (March 5th) Professor Walter Raleigh will begin a course of three lectures on "English Letter Writers." The Friday Evening Discourses this week (Feb. 25th) will be delivered by Captain Abney, C.B., F.R.S., whose subject will be "The Theory of Colour Vision applied to Modern Colour Photography." On March 4th Professor T. E. Thorpe, F.R.S., will deliver the Discourse; his subject is "Some Recent Results of Physico-chemical Inquiry."

MEETINGS FOR THE WEEK.

MONDAY, 28th.—Society of Arts, 8. (Cantor Lectures). "The Principles of Design in Form," by Hugh Stannus, F.R.I.B.A.

TUESDAY, March 1st.—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.

WEDNESDAY, 2nd.—Society of Arts, 8. "Kites—their Theory and Practice," by Captain B. F. S. Baden-Powell.

THURSDAY, 3rd.—Chemical, 8. "Note on the Preparation of Dry Hydrogen Cyanide and Carbon Monoxide," by J. Wade, B.Sc., and L. C. Panting, M.B. "Production of some Nitro- and Amido oxy-lutidines," by J. N. Collie, Ph.D., F.R.S., and T. Tickle. "Production of some Nitro- and Amido oxy-lutidines" (Part II.), by J. N. Collie, Ph.D., F.R.S., and Miss L. Hall. "The Interaction of Magnesium and Solution of Copper Sulphate," by E. Divers, M.D., F.R.S.

FRIDAY, 4th.—Royal Institution, 3. "Recent Researches in Magnetism and Diamagnetism," by Prof. J. A. Fleming, M.A., D.Sc., F.R.S.

SATURDAY, 5th.—Royal Institution, 3. "English Letter Writers," by Professor Walter Raleigh, M.A.

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CONTENTS.

ARTICLES:—	PAGE
On Fehling's Solution, by Otto Rosenheim, Ph.D., F.C.S., and Philip Schidrowitz, Ph.D., F.C.S.	97
On the Separation of Thorium, and the Earths of Cerite, by G. Wyrouboff and A. Verneuil.	97
Testing of Formaldehyd, by Carl E. Smith	98
PROCEEDINGS OF SOCIETIES:—	
CHEMICAL SOCIETY.—Observations on the Influence of the Silent Discharge of Electricity on Atmospheric Air—Some Lecture Experiments—The Ultra-violet Absorption Spectra of some Closed Chain Carbon Compounds—Note on the Absorption Bands in the Spectrum of Benzene	100
PHYSICAL SOCIETY.—Observations on the Peak of Tenerife—A New Theory of Geyser Action	103
NOTICES OF BOOKS.—Quantitative Chemical Analysis—The Analysts' Laboratory Companion—A Graduated Course of Chemical Problems	104
CORRESPONDENCE.—The Atomic Weight of Boron	104
CHEMICAL NOTICES FROM FOREIGN SOURCES	105
MISCELLANEOUS	106
MEETINGS FOR THE WEEK	106
NOTES AND QUERIES	106

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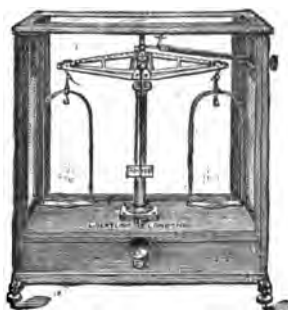
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THE CHEMICAL NEWS.

Vol. LXXVII., No. 1997.

ON FEHLING'S SOLUTION.

By OTTO ROSENHEIM, Ph.D., F.C.S., and
PHILIP SCHIDROWITZ, Ph.D., F.C.S.

In a recent communication under the above heading (CHEMICAL NEWS, vol. lxxvi., p. 318) we showed that the alkali salts of mineral acids have no influence on the reducing power of Fehling's solution. We were led to investigate the matter in consequence of a paper by M. Z. Jovitschitsch (*Ber. Deutsch. Chem. Ges.*, xxx., 2431), in which it was asserted that Fehling's solution is reduced by mineral acids when the reaction of the solution is still distinctly alkaline.

From the perusal of Jovitschitsch's paper, we—and we should imagine any other reader (see also J. E. Gerock, *Apoth. Ztg.*, xii., 800; and *Ber. Deutsch. Chem. Ges.*, xxx., 2865)—were forced to the conclusion that the reduction is due to the action of mineral acids (in an alkaline solution!) on tartaric acid; more especially as Jovitschitsch's very words are "that the acids mentioned" [in an alkaline solution (authors)] "favour the decomposition of the tartaric acid; in fact, the tartaric acid itself . . . causes reduction."

We refrained from discussing the matter fully in our first note, for reasons then mentioned, and because we considered the figures published therein sufficiently convincing. But as M. Siegfried (on whose observation Jovitschitsch's paper was based) has lately published a note (*Ber. Deutsch. Chem. Ges.*, xxx., 3133), according to which the (supposed) reduction is now ascribed by him—there was no mention of this in Jovitschitsch's paper—to the deficient alkalinity of the solution caused by the addition of the mineral acids in question, we feel induced to publish the remainder of our qualitative and quantitative experiments.

According to Jovitschitsch's somewhat vague statement—which, by the way, has already formed the subject of abstracts in various journals (see *Journ. Soc. Chem. Ind.*, xxvi., 1046; *Journ. Chem. Soc.* 73 and 74, 1898)—the reduction is best carried out as follows:—

"One or two drops of the mineral acid—not too strong (?)—are added to 1–2 c.c. of strongly (?) alkaline Fehling's solution; that is, till the latter is still somewhat blue or slightly alkaline. Then a few drops of the original Fehling's solution are added. A turbidity similar to that formed by grape-sugar, phenylhydrazin, &c., is immediately produced, and on gently warming the solution the reduction thus commenced is completed." (N.B.—We translate literally).

We have made numerous experiments in strict accordance with Jovitschitsch's instructions, but not in a single instance—even after prolonged boiling—did we observe the slightest reduction (cf. also J. E. Gerock, *loc. cit.*; *Centralbl. Zuckerindustrie*, vi., 245). Quantitative experiments with 25 c.c. of freshly-prepared Fehling's solution and HCl, H₂SO₄, HNO₃, respectively, also gave negative results. In each case the solution was boiled for one minute.

In another series of experiments a constant volume of the solution was first acidified by the addition of hydrochloric, sulphuric, nitric, and tartaric acids respectively in slight excess, and then fresh Fehling's solution added till the reaction was distinctly alkaline to litmus. In every instance the solution remained perfectly clear in the cold.* On boiling, however, a slight turbidity, which was

not increased by four minutes' continuous ebullition, was formed. This turbidity was caused by a small quantity of a yellow substance suspended in the liquid. After filtering through a Soxhlet's asbestos tube, the solution was observed to be of the same colour as before, and did not produce any further turbidity on renewed boiling for some minutes. It was evident from the figures obtained before and after oxidation that the yellow substance only consists in part of hydrated cuprous oxide, and contains varying percentages of organic matter (cf. Ed. Nihoul, *Chem. Zeit.*, xvii., 500; xviii., 881). The carbonic acid formed by the combustion of the organic matter in a current of oxygen was identified by passing the gases into baryta. It will be seen from the figures below that the quantity of reduced copper (after heating in oxygen and subsequently in hydrogen) is so small that it is practically negligible; it is apparently constant and independent of the quantity of solution used.

Jovitschitsch fears that the reduction mentioned above may have been a frequent source of error in the qualitative examination of liquids with Fehling's solution, but we consider this is out of the question. When using *pure* Fehling's solution, the turbidity formed is so slight that it could hardly deceive the veriest novice. It is, of course, clear that we do not wish to recommend the omission of the traditional rule that all liquids to be examined with Fehling's solution must first be made alkaline.

The following were the figures obtained by boiling 25, 50, 100, and 200 c.c. respectively, after adding acids, as described above:—

Acid used.	25 c.c. Fehling's solution.		50 c.c. Fehling's solution.	
	Weight of precipitate.		Weight of precipitate.	
	Before oxidation and reduction.	After oxidation and reduction.	Before oxidation and reduction.	After oxidation and reduction.
Tartaric acid ..	0.0022	0.0007	0.0027	0.0009
Sulphuric acid .	—	0.0002	0.0022	0.0006
Nitric acid ..	0.0020	0.0010	0.0016	0.0002
Hydrochloric acid ..	—	0.0010	0.0020	0.0002
	100 c.c. Fehling's sol.		200 c.c. Fehling's sol.	
Sulphuric acid .	0.0017	0.0006	0.0025	0.0009

ON THE SEPARATION OF THORIUM, AND THE EARTHS OF CERITE.

By G. WYROUBOFF and A. VERNEUIL.

AFTER having found a method for the separation of cerium from lanthanum and didymium, and the whole of the earths of the yttria group, we sought for a more convenient and more certain method than those yet known for getting rid of the thorina. In all the reactions made use of for the separation of the cerite earths, thorium remains partly, if not entirely, with the cerium. This occurs particularly in the method of separation which we have already described. If only a few hundredths are present, they are completely precipitated at the same time as the cerocerite nitrate.

If we require simply to obtain pure cerium, we dissolve the precipitate in a sufficient quantity of hydrochloric acid to reduce the cerium to the state of protoxide, add a little phosphoric acid to the warm solution, and evaporate to a pasty consistency; then take up with water, and

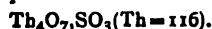
* M. Siegfried (*loc. cit.*) has modified Jovitschitsch's original communication by stating that reduction does not take place till the liquid is boiled.

* It is even smaller than the quantity obtained by boiling dilute Fehling's solution with or without the addition of alkali salts (cf. CHEM. NEWS, vol. lxxvi., 318).

filter. All the thorina remains in the insoluble state, separating a few hundredths of cerium. The filtered liquid contains protochloride of cerium, perfectly free from thorium.

The inverse problem—the preparation of thorina perfectly free from cerium—is much more difficult, and, as is well known, it can only be achieved after a long series of operations, without which we can obtain no precise characteristic, except the absence of incandescence, for the purpose of recognising its purity.

Some time ago (*Bull. Soc. Chim.*, xliii., p. 57, 1885) M. Clève announced a curious reaction, which belongs only to thorina among the rare earths: on adding peroxide of hydrogen to a solution of sulphate of thorium we obtain a gelatinous precipitate which has for formula—



By studying this reaction more closely we observed that by using the nitrate as exempt from the free acid as possible, and adding an excess of H_2O_2 and heating to about 60° , we precipitate the whole of the thorium present in the solution. The reaction is so sensitive that we can not only detect one part of thorium in a thousand of either cerium or of the mixed earths of cerite and gadolinite, but we can even collect and weigh it. It is true that the thorina thus precipitated still contains some cerium, especially if this latter is present in great excess. But by re-dissolving this impure thorina in nitric acid, evaporating to dryness, and precipitating once more with H_2O_2 , we can obtain a thorina containing not more than 0.1 per cent of impurities. A third precipitation removes these last traces of cerium, and after a fourth precipitation the filtered liquid does not give the slightest precipitation with ammonia. In practice, and especially if working on several hundred grms. of material, it is better to commence by treating the mixture of nitrates with an excess of a 10 per cent solution of carbonate of ammonia to which a little caustic ammonia has been added. All the thorina thus passes into solution, carrying only a few hundredths of the other earths with it. A single precipitation with peroxide of hydrogen then suffices to obtain a precipitate of thorina almost perfectly pure, and giving only a trace of incandescence.

It remained for us to apply this simple and rapid method to the quantitative estimation of thorium in the presence of the other rare earths; but here altogether unexpected difficulties were met with. The nitrate of the peroxide, $\text{Th}_4\text{O}_7\text{N}_2\text{O}_3$, cannot be calcined, as in losing its oxygen it decrepitates, and is reduced to a powder of such extreme tenuity that it is thrown right out of the crucible, which may easily lead to a loss of 10 per cent. On the other hand, it is impossible to dissolve it on the filter, in either nitric or hydrochloric acid, so as to transform it into hydroxide, the calcination of which is easy; in fact, such a considerable disengagement of oxygen takes place that, whatever precautions one takes, a certain quantity of the solution is carried off in imperceptible drops. After a large number of attempts we decided on the following method, which, as will be seen further on, gives perfectly satisfactory results. The solution of the nitrates, which should not contain more than 0.5 of oxides, is evaporated to dryness, and taken up with 100 c.c. of water and 10 c.c. of peroxide of hydrogen,* and heated for some minutes while stirring. The extremely voluminous precipitate is thrown on a filter and washed until the wash water no longer gives a precipitate with ammonia. The precipitate is detached from the filter,—an operation which is not at all difficult, owing to the gelatinous nature of the peroxide,—and dissolved in a few c.c. of water, containing 2 grms. of NH_4I and 2 c.c. of concentrated HCl ; the reduction is instantaneous. The hot solution is run through the filter to dissolve the

proportions of the precipitate still adhering to it, then washed. The solution is precipitated by ammonia; the hydroxide, which does not need washing, is thrown on the same filter and calcined. In the filtered liquid of the peroxide, all the other earths which accompany thorium are precipitated by ammonia. The precipitate obtained by means of peroxide of hydrogen ought to be perfectly white. If such is not the case—which happens whenever cerium is present in any considerable quantity—it is necessary to re-dissolve the hydroxide in nitric acid, evaporate, and repeat the operation. As a general rule, if very exact results are wished for, this double precipitation is strongly to be recommended, as well for the peroxide of thorium, which entangles a little cerium, as for the other earths, which may carry with them a few thousandths of thorina.

	I.		II.		III.	
	Taken.	Found.	Taken.	Found.	Taken.	Found.
ThO ..	0.3670	0.3655	0.3645	0.3640	0.0508	0.0475
Ce ₂ O ₄ ..	0.0231	0.0229	0.0214	0.0370	0.9485	0.9515
LaODiO	—	—	0.0140			

We give the analysis No. III. to show the results obtained by a single precipitation.

M. Dennis (*Zeitsch. fur Ann. Chim.*, xliii., p. 412, 1897) has quite recently proposed the potassic salt of hydronitric acid for the separation of thorina from the other rare earths. This curious reaction, as a matter of fact, permits of the total precipitation of the thorina present in the mixture. Unfortunately it presents two obstacles; viz., it requires the use of a reagent which is not at all convenient to handle in any quantity, and, further, it has the grave fault of carrying down with it several hundredths of cerium which are not eliminated even by a second precipitation. In an artificial mixture containing 0.0952 of ThO and 0.927 of CeO we found, after the precipitation of the thorina by KN_3 , only 0.0896 of CeO. The precipitated thorina had thus carried with it 3.34 per cent of CeO. It was then dissolved in nitric acid and precipitated with peroxide of hydrogen; the filtered solution contained 0.0028 of CeO. This experiment clearly shows that the method we propose is not only more practicable, but also more precise, than the still interesting one proposed by M. Dennis.—*Comptes Rendus*, cxxvi., No. 4.

TESTING OF FORMALDEHYD.*

By CARL E. SMITH.

(Concluded from p. 95).

Acetone.—Three grms. of formaldehyd solution, 25 c.c. of N/1 soda solution, and 0.5 c.c. of pure acetone were heated under pressure in boiling water for forty-five minutes. A duplicate assay, omitting the acetone, was made under the same conditions. In the mixture containing the acetone a white flocculent precipitate formed on heating, and titration with sulphuric acid indicated that not more than one-third of the theoretical amount of soda had been used up. The duplicate containing no acetone gave normal figures. No attempt has yet been made to ascertain the composition of the precipitate and the cause of its formation.

Methyl Alcohol.—To 20 c.c. of a dilute aqueous solution of pure formaldehyd, 1 c.c. of commercial methyl alcohol was added and the mixture heated with soda solution, as described in the acetone experiment. The solution was assayed under the same conditions without addition of methyl alcohol:—

Without methyl alcohol ..	4.65, 4.65, 4.71 per cent.
With " " " "	4.38, 4.35 " "

* Commercial peroxide of hydrogen containing any considerable amount of impurities cannot be used. It must be purified by distillation; this is no longer difficult since the work done by M. Harriot.

* Report of Research Committee DII., Committee of Revision of the United States Pharmacopoeia. From the *American Journal of Pharmacy*, lxx., No. 2.

Repeated with pure methyl alcohol, 0.5 c.c. of this was added to 2.25 grms. of a concentrated solution of pure formaldehyd:—

Without methyl alcohol 37.3, 37.3 per cent.
With " " " " 35.5, 35.7 "

No explanation can be made at present for the lowering of the result by the presence of methyl alcohol.

The chief disadvantages of the fixed-alkali method are interference by acetone and methyl alcohol and risk of explosion when heating under pressure. It is also subject to some variation in the results—noticed with one sample only—the causes of which cannot be satisfactorily explained at present.

Ammonia Method.—First proposed by Legler (*Berichte*, vol. xvi., p. 1333), and based on the reaction of free ammonia and formaldehyd to form hexamethylene-tetramine, $6\text{CH}_2\text{O} + 4\text{NH}_3 = \text{N}_4(\text{CH}_2)_6 + 6\text{H}_2\text{O}$.

A normal ammonia solution is usually recommended, while the decinormal is preferred by others. As the use of the latter necessitates weighing or measuring very minute quantities of formaldehyd, or else diluting a larger quantity and taking an aliquot part for assay, the work was restricted to the use of the stronger solution. Only one trial with a decinormal solution was made, which indicated that the assay would require about one hour, and that rosolic acid is not sufficiently sensitive in presence of hexamethylene-tetramine to give serviceable results until after some practice.

Experiments with N/1 ammonia solution:—25 c.c. of the ammonia solution were run from a burette into a flask and 2.25 grms. of sample added. The flask was then closed with a glass stopper thickly coated with vaseline. The mixtures were allowed to stand a definite time, and then the excess of ammonia titrated with N/1 sulphuric acid, using rosolic acid as indicator. Each c.c. of N/1 ammonia corresponds to 0.045 gm. or 0.5 per cent, when 2.25 grms. are taken.

Titrated at once after admixture 36.0 per cent.
" after 15 minutes 37.4 "
" " 1 hour 37.5 "
" " 2 hours 37.5 "
" " standing over night 37.4 "

Other trials, previously made in flasks closed with rather loosely-fitting rubber stoppers, gave somewhat variable figures, the tendency being too high results through loss of ammonia, particularly when considerable time elapsed before titrating.

Assay in presence of Acetone.—A mixture of 2.25 grms. of formaldehyd solution, 25 c.c. of N/1 ammonia, and 0.5 c.c. of pure acetone was allowed to stand twenty minutes and then titrated. The solution was assayed in a similar manner without addition of acetone.

Without acetone 34.9, 34.7 per cent.
With " " " " 34.8, 35.0 "

Assay in presence of Methyl Alcohol.—A mixture of 2.25 grms. of formaldehyd solution (not the solution used in the preceding experiments), 0.5 c.c. of methyl alcohol, pure or commercial, and 25 c.c. of N/1 ammonia, was treated in the same manner as above, and the formaldehyd solution also assayed without addition.

Without methyl alcohol 37.4, 37.4 per cent.
With pure methyl alcohol 37.5, 37.6 "
" commercial methyl alcohol .. 36.8, 36.9 "

According to these figures, commercial methyl alcohol may lower the result slightly, while pure methyl alcohol and acetone exert practically no influence. This method, then, is the only thoroughly reliable one of all those tried, the only drawbacks being a slight inaccuracy introduced through loss of ammonia by volatilisation, and the necessity of frequently re-standardising the ammonia solution, which loses strength rapidly. These considerations suggested to the writer a modification of this method, which

obviates the need of keeping on hand a standard solution of ammonia, and reduces the volatilisation of ammonia during the manipulation to a minimum.

Ammonia Method Modified.—Dissolve 2 grms. of pure neutral ammonium chloride in 25 c.c. of water and introduce it into a flask provided with a well-fitting stopper. Add 2.25 grms. of the sample, and then run in from a burette 25 c.c. of N/1 potassium (or sodium) hydrate. Stopper the flask at once and put it aside for one-half hour. Then add a few drops of rosolic acid solution and determine the excess of ammonia with N/1 sulphuric acid, each c.c. of N/1 potassium hydrate consumed indicating 0.5 per cent of formaldehyd.

The results obtained by this modification agree closely with those obtained by the hydroxylamine, fixed-alkali, and ammonia methods. The reactions involved are as follows:—



or—



The ammonia combines with the formaldehyd nearly as fast as it is liberated, and, consequently, has no chance to volatilise, and the final excess is so small that the odour is barely perceptible.

Examination of Commercial Samples.

Sample I.—Colourless, contains a white flocculent precipitate, probably para-formaldehyd, which increases on keeping, more rapidly when exposed to diffused sunlight than in the dark. Specific gravity, 1.109 at 15°/15°; free acid, assumed to be formic acid, 0.17 per cent; residue on ignition, 0.19 per cent, consisting of sodium chloride and a little sodium carbonate.

Strength, by ammonia method .. 37.4, 37.5 per cent.
" fixed-alkali method .. 37.3, 37.6 "
" hydroxylamine method 37.2, 37.3 "

Sample II.—Colour faintly yellowish; contains fine particles of extraneous matter, but is free from a deposit of para-formaldehyd. Specific gravity, 1.052 at 15°/15°; free acid, 0.065 per cent; traces of acetone; residue on ignition, 0.015 per cent, consisting of copper oxide.

Per cent.

Strength, by ammonia method .. 34.6, 34.7
" fixed-alkali method .. 35.3, 34.7, 35.6, 34.0
" hydroxylamine method .. 34.4, 34.6

Cause of discrepant results by the fixed-alkali method unknown. This solution is claimed to be 40 per cent strong on the label.

Sample III.—Clear, faintly yellowish; specific gravity, 1.057 at 15°/15°; free acid, 0.07 per cent; traces of acetone; residue on ignition, 0.032 per cent, consisting of copper oxide.

Strength, by ammonia method (modified) 35.3, 35.2 p. c.
" fixed alkali method .. 35.3, 35.2 "
" hydroxylamine method .. 35.0, 35.1 "

Samples II. and III. are apparently made directly from wood-spirit, as judged by the presence of acetone, copper, and by the disproportionately lower specific gravity, indicating presence of a lighter liquid, probably methyl alcohol. These samples also have more empyreuma than is the case with Sample I.; they leave a smaller residue of para-formaldehyd when evaporated spontaneously and deposit no para-formaldehyd when exposed to daylight for nearly a month.

Tests of Identity and Purity.

The following list of tests is presented as a convenience to those who may wish to avail themselves of it as a guide in the examination of commercial products. The

details of the tests are arranged in such a manner as experiments seemed to show most practical. No test for methyl alcohol is included, as it was not found practicable to test for it in presence of formaldehyd except by tedious distillation. The pungent odour of formaldehyd completely masks the wintergreen odour obtained by the salicylic acid test, even when a large quantity of methyl alcohol is known to be present.

The solution should contain 35 to 40 per cent of absolute formaldehyd, as ascertained by the ammonia method or its modification.

It should be transparent and colourless, have a pungent odour and caustic taste, and a neutral or faintly acid reaction. Specific gravity about 1.08 at 15° C. Miscible in all proportions with water and with alcohol. On mixing with an ammoniacal solution of silver nitrate, metallic silver is separated. Heated with alkaline copper tartrate solution, cuprous oxide is separated.

If to 2 c.c. of the solution an equal volume of potassa solution and about 0.5 grm. of resorcinol be added and the mixture heated to boiling, the yellow colour which first appears gradually becomes red. (This reaction is said to be given by no other substance).

If 5 c.c. of sulphuric acid (specific gravity 1.84) be placed in a test-tube with a little salicylic acid and two drops of formaldehyd solution added, a permanent deep red colour will appear immediately.

If 1 c.c. of the solution be evaporated to dryness on a water-bath after addition of 5 c.c. of ammonia water, a white crystalline residue will remain, which, upon moistening with dilute sulphuric acid and warming, will evolve the pungent odour of the original solution. (Re-conversion of hexamethylene-tetramine into formaldehyd and ammonia).

If 5 c.c. be evaporated to dryness on a water-bath, a white amorphous mass is left, which should leave no residue on ignition. (Absence of mineral impurities).

Ten c.c. should require not more than 0.25 c.c. of N/1 potassium hydrate for neutralisation, using phenolphthalein as indicator (absence of more than 0.1 per cent of formic acid).

A coil of clean platinum wire dipped into the solution and held into a non-luminous flame should not colour the flame yellow (absence of sodium), nor should it appear violet when viewed through a blue glass (absence of potassium).

Dilute the solution with three times its volume of water for the succeeding tests. No turbidity or precipitate should be caused by silver nitrate (absence of chloride); nor by barium chloride (sulphate), nor by hydrogen sulphide or potassium ferrocyanide (metals), nor by ammonium oxalate (calcium).

If 1 c.c. of formaldehyd solution be mixed with 10 c.c. of iodine test solution, and potassa solution added until the solution becomes colourless, no precipitate should be formed, nor an odour of iodoform developed (absence of acetone).

The solution should be kept in dark amber-coloured bottles, in a cool place, protected from light.

The writer is much indebted to Prof. Virgil Coblentz, in whose laboratory this work was carried on, for his great interest in these investigations, and many valuable suggestions in connection with them.

Lining Iron Pipes.—We are informed that Messrs. Henderson, Kandt, and Co., Ltd., have succeeded in coating the interior of iron pipes of small diameter with a smooth and perfect layer of porcelain enamel, and they are placing them on the market under the name of "Perlam Conduit." This announcement will be of interest to many of our readers who have hitherto been accustomed to use glass-lined tubes for the conveyance of corrosive fluids.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 17th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. George F. Merson and Ernest H. Roberts were formally admitted Fellows of the Society.

STATEMENT BY THE COUNCIL.

The PRESIDENT said he was authorised by the Council to make the following statement to the Society:—

The following letter has been received by the Council:—

Letter received by the President from Messrs. Harden and Hartog.

The Owens College, Manchester, Jan. 26th, 1898.

DEAR SIR,—We beg to acknowledge the receipt of your letter of the 22nd inst. with reference to the Memorial which we had the honour of transmitting to yourself as President, and to the Council, of the Chemical Society, and to offer you our thanks in the name of the Memorialists for the consideration which it has already received.

We desire to point out that, although it was not expressly so stated in the Memorial, it was obviously the wish of the signatories, if the Council had not at present the power to propose an alteration of Bye-law V. in the sense suggested, that it should take the steps necessary to obtain that power. We gratefully recognise, therefore, the further consideration which the Council is now giving to this aspect of the question.

Would it be possible for us to be informed if it is the intention of the Council to make a definite statement with regard to the questions raised in sufficient time for the matter to be brought before the Anniversary Meeting for this year.—We are, dear Sir, your obedient servants,

ARTHUR HARDEN.
PHILIP HARTOG.

Professor James Dewar, F.R.S., &c.,
President of the Chemical Society.

The following reply had been sent that evening:—

Answer of Council.

Chemical Society, Burlington House, W., Feb. 17, 1898.

DEAR SIR,—The Council desire me to thank you for your letter of the 26th January.

Since the presentation of the Memorial and the receipt of your letter of the 16th December, the question of the Society applying for a Supplemental Charter to enable votes at General Meetings to be given by proxy has received the anxious consideration of the Council, and in view thereof the Council has taken further legal advice on the matter.

The Council has this evening directed the President to make an announcement to the Society, which will be printed in the next *Proceedings*.—I am, dear Sir, your obedient Servant,

JAMES DEWAR, President.

Messrs. Harden and Hartog.

Report of the Council.

The Council has submitted the question of a Supplemental Charter for legal opinion. The following was the case submitted:—

Case for the Opinion of Council.

On the 6th December last Council advised the President and Council of the Chemical Society whether under the Charter of such Society they had power to alter the Bye-Laws so as to provide for the Votes at General Meetings being given by proxy. The case then laid before Council and his opinion thereon are sent herewith.

The Memorial referred to in the case has now been presented to the President and Council. It was signed by over 500 Fellows, or about one-quarter of the total number. A copy thereof is sent herewith.

Together with the Memorial, a letter dated the 15th December, 1897, was received from Mr. Arthur Harden and Mr. Philip Hartog, a copy of which is also sent herewith.

Such letter contains the following passage:—

With reference to the substance of the Memorial we wish to make one statement. We are aware that in the Charter (on page 8 of the printed text) it is provided that "at all general meetings and meetings of the Council the majority present and having the right to vote thereat respectively shall decide upon the matters propounded at such meetings" hence in order to render legal the change desired it would probably be necessary to obtain a Supplemental Charter allowing members not actually present at meetings to vote by means of balloting papers. It is in this connection material to point out that in order to obtain a revision of Bye-Laws precisely similar to that now desired by Fellows of our own Society a Supplemental Charter was quite recently obtained by the Institution of Civil Engineers:

It is upon the question of a Supplemental Charter that the opinion of Council is now desired.

Enquiries have been made with reference to the Supplemental Charter obtained by the Institution of Civil Engineers referred to in the above-mentioned letter. Such Supplemental Charter was obtained in March, 1896, and a printed copy thereof together with a copy of the original Charter and Bye-Laws of such Institution is sent herewith.

It appears that the circumstances under which that Supplemental Charter was granted were not analogous to the facts connected with the Chemical Society, and it is questionable whether that case can be regarded as a precedent in favour of granting a Supplemental Charter to such Society provided the Society as a whole should decide to present a petition for one.

In the first place the members of the Institution of Civil Engineers were unanimous in desiring the change. In the present case it is believed that the Fellows are not unanimous on the question, and it is not at present known whether even a majority desire the change.

Secondly, the objects of the Chemical Society and of the Institution of Civil Engineers respectively are wholly different.

The object of the Chemical Society shortly stated is the general advancement of Chemical Science by the holding of Meetings at which new discoveries are brought under discussion and the results made known to the public in a series of *Transactions*.

It is a purely scientific Society and in no sense professional, and in this respect resembles rather the Royal Society, the constitution of which requires the presence of Fellows at general meetings to enable them to vote.

The object of the Institution of Civil Engineers, on the other hand, is not merely for the general advancement of Mechanical Science; it is "more particularly for promoting the acquisition of that species of knowledge which constitutes the profession of a Civil Engineer." It holds examinations for the admission of persons desirous of becoming Associate Members, and is to a large extent a body exercising administrative functions with respect to a particular profession all over the Kingdom. Obviously therefore the considerations which would induce the Authorities to vary the regulation and management of such a body as the Institution of Civil Engineers would not be applicable in the case of a purely Scientific Society.

Thirdly, the reasons advanced for granting the Supplemental Charter to the Institution of Civil Engineers do not exist in the case of the Chemical Society. The

Institution of Civil Engineers stated in the Petition for such Supplemental Charter whereas when their Charter was granted (in 1828) the number of Members of all classes was 156, that—

On the 2nd day of January 1896 the number of Members of all classes was 596 the prescribed number of members of the Council is no longer sufficient to enable the Institution to place on the Council an adequate number of representative men having regard to the total number of members and the scope of all branches of engineering science as now taught and practised throughout the realm and the number of members residing in or near the Metropolis now forms a comparatively small portion of the whole number and a large proportion cannot now be reasonably expected personally to attend General Meetings &c. &c.

In the case of the Chemical Society it cannot be alleged that the number of the Members of the Council is inadequate having regard to the total number of the Fellows, the present number being 37 (including 15 vice-presidents who have been presidents) out of a total number of about 2100 Fellows. Under the existing Bye-Laws of the Institution of Civil Engineers the number of the Council "shall not be less than 23 nor more than 31," the total number of members of all classes of such Institution being at the present time about 6900.

It cannot be alleged that the Council of the Chemical Society has not contained from its foundation an adequate number of representative men. In fact the Memorial in favour of an alteration in the bye-laws states:—

It is of course recognised that a custom has grown up of choosing a certain proportion of the officers and Council from among the Fellows who reside in the provinces.

It cannot be alleged that the number of Fellows of the Chemical Society residing in or near the Metropolis forms a comparatively small portion of the whole number, or that a large proportion cannot now be reasonably expected personally to attend General Meetings. There has not in recent years been any material increase (see Table of Statistics below) in the proportion of the number of Fellows who do not reside in or near the Metropolis as compared with the whole body, and there is the further fact that whereas the object of the Society above mentioned has remained unchanged since the date of its charter in 1849, means of transit and communication with the other parts of the kingdom have since that date become greatly facilitated.

Statistics of the Society.

	No. of Fellows.	Resident in London District.		Not resident in Great Britain and Ireland.		Country Members.	
		Per cent.		Per cent.		Per cent	
1869	526	241	45	27	5	258	49
1871	550	255	46	36	5	269	50
1881	1090	461	42	113	10	516	48
1891	1840	625	34	289	16	926	50
1897	2100	700	33	322	16	1078	51

So far as is known there is no case in which a Supplemental Charter has been granted to an old scientific Society on the question of voting by proxy alone, where that question has not involved other considerations than the mere question of attendance of the members thereof at General Meeting, even if such body was unanimous in desiring a change in its constitution for this purpose.

Council is requested to advise the President and Council of the Chemical Society as to the chances of success of obtaining a Supplemental Charter.

- 1 If the Fellows were unanimous.
- 2 If there were a majority of the Fellows in favour of the proposed change, and the minority took no active steps to oppose.
- 3 If there should be active opposition on the part of a minority of the Fellows.

- 4 If there were only a minority of the Fellows in favour of the change.
- Also 5 How the majority for or against the change is to be ascertained. Must it be a majority of the whole of the Fellows or a majority of those voting at a General Meeting.

The following is the opinion of Counsel.

Copy of Opinion of Mr. Cozens-Hardy, Q.C.

Opinion.

1, 2, 3, and 4. The questions raised in this case are not really questions of law, and I do not feel that any opinion I can give as to the chances of success of obtaining a Supplemental Charter can be of any value. But I may say that I think it highly improbable that the Government department, before whom the application must come, would be disposed to listen to the application unless it represented the practically unanimous view of the Fellows, and that any active opposition by even a small minority would probably be fatal. The propriety of allowing votes by proxy, or by voting papers transmitted through the post, is obviously open to reasonable doubt. It is not easy to alter the constitution of a body of this nature, except to give effect to the practically unanimous wish of the Corporators. I may add that the Supplemental Charter granted in 1895 to the Institution of Civil Engineers does not really furnish a precedent applicable to the present case. That charter dealt with various important matters of which the allowance of proxy votes or voting papers was only one. I understand also that there was no difference of opinion on the part of the Fellows of that Institution.

5. In considering whether the Fellows really desire the change proposed, I think regard must not be had only to those who are present in person at a meeting, but in some other way the wishes of the whole body must be ascertained.

HERBERT H. COZENS-HARDY.

7, New Square, Lincoln's Inn,
February 4, 1898.

It was announced that the following changes in the Officers and Council were proposed by the Council:—

As *Vice-Presidents*—Professor Liveing, F.R.S., and Professor J. M. Thomson, F.R.S., *vice* Mr. Ludwig Mond, F.R.S., and Professor Roberts-Austen, C.B., F.R.S.

As *Hon. Secretary*—Dr. W. P. Wynne, F.R.S., *vice* Professor J. M. Thomson, F.R.S.

As *Ordinary Members of Council*—Messrs. E. J. Bevan, H. J. Fenton, W. Gowland, and David Howard, *vice* Messrs. B. H. Brough, J. W. Rodger, T. K. Rose, and Professor Sydney Young.

Certificates were read for the first time in favour of Messrs. Albert Abbot, B.A., Church Street, Adlington, Chorley; Henry William Coupe-Annable, University College, Sheffield; William Thomas Gidden, 108, Vicarage Road, Langley, Birmingham; Alexander Guthrie, B.Sc., Bocking, Braintree, Essex; John Heaton, 81, Garroyle Road, Liverpool; John Shearson Hyland, Ph.D., M.A., 11, Powis Square, Bayswater; William Jones, 29, High Street, Wavertree, Liverpool; Thomas Martin Lowry, B.Sc., 28, St. Lawrence Road, N. Kensington, W.; Albert Henry Mitchell, Martin's Lane, Tiverton; Alfred James Parker, 21, East Hill, Dartford, Kent; Samuel Walker, M.A., B.Sc., 126, Gilmore Place, Edinburgh; Samuel Allinson Woodhead, B.Sc., Agricultural College, Uckfield.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected:—

Messrs. Ernest Lionel Allhusen, B.Sc.; William Martin Bailey; Charles Edward Brittain, B.Sc.; Cecil Joslin Brooks; Benjamin S. Bull, M.A., B.Sc., Ph.D.; Charles Henry Burge; William Arthur Caldecott, B.A.; Matthew J. Cannon; Albert John Bullen Cooper; John Cooper, B.Sc.; William R. Cooper, M.A., B.Sc.; Frederick Cowling; Frederick Robertson Dodd; John R. Don,

D.Sc., M.A.; Frederick W. Dootson, M.A.; Wilbraham T. A. Edwards; Frederick Gilderdale; William Setten Gilles; John Glaister, M.D.; Thomas Herbert Hills; David Homfray, B.Sc.; Atholl Francis McEwen; William Hobson Mills, B.A.; Gilbert Thomas Morgan; William Edward Moss; Herman Poole; Thomas Henry Pope; James Brown Reid; Frank Forster Renwick; William Colebrook Reynolds; William Richards; Harold Charles Sayer; Andrew Jamieson Walker, B.A.; Ernest Charles Weissmüller.

Messrs. H. T. Brown, F.R.S., F. D. Chattaway, and R. J. Friswell were appointed to audit the Society's accounts.

Of the following papers those marked * were read:—

* 14. "*Observations on the Influence of the Silent Discharge of Electricity on Atmospheric Air.*" By W. A. SHENSTONE and W. T. EVANS.

When air is submitted to the action of the silent discharge it first contracts to a remarkable extent, and then re-expands rapidly until it very nearly occupies its original volume. The residue contains a trace of nitric peroxide. The following are some of the chief conclusions arrived at from a study of the above phenomena. Oxygen, when diluted in nitrogen, as in the air, yields a very large proportion of ozone; 80–85 per cent of the oxygen present may readily be ozonised in the presence of moisture, and if great care be taken as much as 98 per cent of the oxygen may be converted into ozone.

If the ozonising of the oxygen be not pressed too far no nitric peroxide will be formed, but at a certain stage, which probably coincides, or nearly coincides, with the point at which the amount of ozone is at its maximum, nitric peroxide is formed. In the presence of nitric peroxide, ozone is rapidly destroyed by the silent discharge, and its destruction is accompanied by a considerable destruction of nitric peroxide. The presence of water vapour promotes the formation of ozone, but retards that of nitric peroxide. It was found to be impossible to ozonise the oxygen of air in the presence of a trace of nitric peroxide.

DISCUSSION.

Dr. SCOTT asked if the diminution in the quantity of nitrogen peroxide present, when all, or almost all, the ozone had again been decomposed, might not be explained by its conversion by the moist air into nitric acid, and so overlooked by the method of estimating the oxidised nitrogen which would not recognise it in this form.

Mr. SHENSTONE, in reply, said that he had not studied the effect of disruptive discharge, nor that of other diluents. In reply to Dr. ARMSTRONG, he said that the electrical conditions had been carefully regulated in the manner described in a previous communication to the Society, but all such details were now omitted in order to economise space. In the present experiments, the difference of potential at the "spark gap" was about 13,300 volts. With regard to the question asked by Dr. Scott, of course one could not be sure that no nitric acid had been formed when the nitric peroxide disappeared in the presence of water, but this could hardly have occurred with the dried gas, and the behaviour of dried and damp gases seemed to correspond. Moreover, the gas was always re-measured after the ozone had been destroyed, and the amount of permanent contraction did not support the idea that the nitric peroxide had been to any considerable extent converted into nitric acid.

* 15. "*Some Lecture Experiments.*" By J. TUDOR CUNDALL, B.Sc.

Conservation of Mass.—Phosphorus in a weighed and closed flask is set on fire by passing a hot wire down the hollow stem of the deflagrating spoon containing it, thus raising the temperature of the spoon to the igniting point of the phosphorus.

Graham's Law of Diffusion.—A tube is filled so that it can be filled with hydrogen and other gases, and then

connected to a filter pump by means of a closed up piece of the stem of a clay pipe, through the walls of which the gas can diffuse into the partial vacuum. The times that it takes for water to rise from one mark to another when the tube is filled with different gases are noted, and it is found that these are very nearly in the same ratio as the square roots of the densities of the gases.

DISCUSSION.

Professor ARMSTRONG observed that the apparatus used for ignition of the phosphorus was needlessly complicated. All that is necessary is a glass flask, in which a piece of phosphorus is ignited by heating the flask, which is then closed with a rubber stopper. In this simple form the experiment was well known in many elementary schools throughout the country.

* 16. "Note on the Preparation and Properties of *O*-Chlorbrombenzene." By J. J. DOBBIE, M.A., D.Sc., and FRED MARSDEN, M.Sc., Ph.D.

In this paper the authors describe the preparation and properties of *o*-chlorbrombenzene. It is a clear straw-coloured liquid with a strong aromatic odour. It boils constantly at 204° (i V) under a pressure of 765 m.m., and does not solidify at -10°. Sp. gr. at 12.5° = 1.6555 μ_D = 1.583.

* 17. "The Ultraviolet Absorption Spectra of some Closed Chain Carbon Compounds." By W. N. HARTLEY, F.R.S., and J. J. DOBBIE, M.A., D.Sc.

The authors give the results of the examination of the absorption spectra of a number of organic substances differing in constitution from those previously examined by Professor Hartley and his fellow workers. The absorption spectra of substances containing closed chains, wherein four or six carbon atoms are singly linked, or two pairs are doubly linked, and of substances containing oxygen or other polyvalent atoms as links in a closed chain, were studied. The examples selected were diketohexamethylene, pyrrol, thiophene, furfuran, furfural, pyromucic acid, and furfuramide. Tables showing measurements of the extent and intensity of the absorption by each of these substances are given. The absorption of the ultraviolet rays by solutions of some of them, and notably of thiophene, is very intense, but in no case is there any selective absorption.

The conclusion arrived at is that however intense the absorption of the ultraviolet rays may be, bodies possessing the constitution of those examined show no absorption bands.

* 18. "Note on the Absorption Bands in the Spectrum of Benzene." By W. N. HARTLEY, F.R.S., and J. J. DOBBIE, M.A., D.Sc.

In the earliest paper on the spectrum of benzene (Hartley and Huntington) it was described as showing six absorption bands, four of which are more persistent and stronger than the other two. Owing to the want of means to obtain a continuous spectrum, some difficulty was experienced in measuring with accuracy the weaker bands. By using a wide slit, lenses of long focus and a powerful spark, the rays are sufficiently continuous, but at a sacrifice of accurate measurement of the fiducial spark lines. One feature, however, was noticeable in spectra obtained in this way: the absorption bands were all degraded in the direction of the least refrangible rays. This was considered as suggestive of the bands consisting of groups of lines stronger and closer together on the more refrangible side, weaker and wider apart on the less refrangible. By using a very powerful spark the rays emitted by cadmium electrodes may be rendered sufficiently continuous to show these bands, and at the same time render the lines sufficiently sharp to measure them by, and from such a photograph, taken a year ago, the absorption bands have been again measured.

The results of the series of measurements taken from this photograph are compared with those obtained by Pauer by examining the vapour of benzene (*Wied. Ann.*,

1897, lxi., 362), and it is shown that his weak line λ 2670 is identical with the first absorption band, and his weak band λ 2390—2360 with the sixth absorption band observed in solutions of benzene in alcohol.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, February 26th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

THIS meeting was held at Eton College.

The PRESIDENT informed the Society of the resignation of one of its Hon. Secretaries, Mr. T. H. Blakesley, M.A. In doing so, he referred to the many important services rendered to the Society by Mr. Blakesley, and he expressed the Society's deep and general regret that Mr. Blakesley should now feel unable to continue them.

The Council elected Mr. W. Watson, B.Sc., to the office of Hon. Secretary.

Prof. T. C. PORTER, in whose laboratory the meeting was held, said it gave him very great pleasure to welcome the Physical Society. Eton had been most properly called "the English home of ancient classical learning." For the education of youth classics had proved itself of cardinal value. He believed that other Fellows of the Physical Society, with himself, desired that this revered tradition of classics should be maintained at Eton. At the same time they would agree with him that there was no better supplement to classics than a fair knowledge of the natural sciences.

Prof. PORTER then gave a lecture, illustrated by lantern photographs, on "*Observations on the Peak of Tenerife*." He also described his method for measuring the diameter of the earth.

The method consists in observing the shadow cast by the Peak upon the sea, and measuring the time that elapses between the moment when the apex of the shadow touches the sea-horizon, and the instant when it is eclipsed by the shadow of night.

Prof Porter called attention to a phenomenon hitherto unnoticed, i.e., that the heated air ascending from the Peak casts a shadow, seen as a faint prolongation of that of the Peak; it rises obliquely from its apex. A photograph was exhibited, taken on a quarter-plate, in which is visible the curvature of the horizon as viewed from the altitude of the Peak.

An interesting series of unique photographs, illustrating the conformation of the Peak and the phenomena of sunrise and twilight in that latitude, was also shown. In regard to twilight, he noticed that the first approach of night, as observed looking eastward, is marked by a dark border of about five degrees width, followed by a sky somewhat lighter.

The lecturer discussed also "*A New Theory of Geyser Action*."

The theories of Bunsen and others fail to explain why the geyser throat appears almost completely full at the end of an eruption. This immediate re-filling is the more remarkable when it is remembered that some of the geysers of the Yellowstone region discharge a million and a half gallons at each eruption, and that the eruptions may occur at five-minute intervals. Moreover, the theories generally accepted assume steeper temperature-gradients than those in a region like Yellowstone. Prof. Porter suggests that the phenomena are better explained on the assumption of an arrangement of strata such as exists in artesian-well districts; the throat or shaft of the geyser being in the position of a well communicating with a subterranean stream—the "tube" of the geyser. From the disturbed nature of the region, the tube of the geyser follows a waved course. The "shaft" rises from the

crest of the terminal wave; the other crests may be steam-traps. Since a basin-like formation is characteristic of all geyser regions, it is fair to assume that the end of the tube remote from the shaft has an outcrop in the hills that form the sides of the basin. By means of this outcrop, water continually flows into the tube. Where the tube does not sink deeply enough to attain the temperature necessary for the generation of steam, a quietly-flowing hot spring is the result. But if at any point the tube descends to underground temperatures sufficiently great, steam is formed, and is trapped at the highest point of a bend. Ultimately this steam checks the flow of water, until the accumulated head of cool water from the hills overcomes the resistance, condenses the steam, and re-establishes liquid continuity. Urged by the pressure behind it, the stream is impelled towards the geyser throat; it forces the hot water before it until equilibrium is once again restored in the tube.

Prof. PORTER afterwards exhibited a Method for Viewing Lantern Projections in Stereoscopic Relief.

A slotted disc rotates in front of two lanterns. These project two stereoscopic views in rapid alternation upon a screen, in such a way that the two projections are approximately superposed. In the rim of the disc other slots are cut through which the observer looks. The arrangement of slots is such that the right or left eye is only able to see the screen at the moment when its own picture, *i.e.*, the picture from the right or left lantern, is on the screen. When the rotation is sufficiently rapid, the views appear as one, without "flicker," in stereoscopic relief.

The PRESIDENT proposed votes of thanks, and the meeting was adjourned until March 11.

NOTICES OF BOOKS.

Quantitative Chemical Analysis; adapted for Use in the Laboratories of Colleges and Schools. By FRANK CLOWES, D.Sc. Lond., F.C.S., and J. BERNARD COLEMAN, A.R.C.Sc. Dublin, F.C.S. Fourth Edition. London: J. and A. Churchill. 1897. Pp. 583.

STILL further revisions, additions, and improvements have been made in this, the fourth edition of Messrs. Clowes and Coleman's well-known book. The subject-matter is divided into seven principal parts, the first five of which are again subdivided into sixteen sections. Part I., containing three sections, naturally begins at the beginning of quantitative work, and here we find full descriptions of the apparatus required, and the general rules, methods, and processes in use for the purpose of making quantitative estimations, and the calculation and entry of results. Part II. contains only one section, and is devoted entirely to simple gravimetric estimations. In Part III., divided into four sections, we read all about volumetric analysis; the apparatus and general methods are first described; after which we come to more detailed and special processes, such as alkalimetric and acidimetric methods, estimations by oxidation or by reduction, &c. Part IV., on general quantitative analysis, has four sections, but they cover a great deal of ground, and extend to over 200 pages. A good deal of attention is paid to water analysis, but we are surprised to find that the well-known and necessary combustion process for estimating the organic carbon and nitrogen in water, devised by Sir E. Frankland and Prof. Armstrong, although described as "the most accurate and trustworthy of these processes," is simply passed over, and the student referred to some other books; it is necessary for everyone having to do with modern water analysis to be thoroughly acquainted with this process, and we see no more reason why the student should be referred elsewhere for this information than for anything else in these pages.

Part V. is on the volumetric estimation of gases, and consists of four sections, in which all the best known methods are described. Part VI. contains a number of reference tables; while Part VII. finishes with a few odds and ends, such as the use and preparation of compressed gases, lists of apparatus, &c.

The revised atomic weights, as re-calculated by Mr. F. W. Clarke for his work which is about to be published, have been obtained in advance, and appear in par. 774.

We congratulate the authors on having given us an eminently useful and practical volume, and we wish them still further success.

The Analysts' Laboratory Companion. By ALFRED E. JOHNSON, A.R.C.S.I., F.I.C. Second Edition, enlarged and improved. London: J. and A. Churchill. 1897. Pp. 108.

A NUMBER of additions and improvements to this very aptly named book have been made in this edition, amongst which may be mentioned the replacing of the tables of seven-figure logarithms by those of five figures. The section devoted to weights and measures has been entirely re-written, as also have the pages dealing with the specific rotatory and cupric-reducing powers of the carbohydrates. The butter analysis and glycerin tables are also new.

The whole book abounds with useful and conveniently-arranged information, and is well interleaved with blank ruled sheets for private or additional notes.

A Graduated Course of Chemical Problems. By E. G. BRYANT, B.A., B.Sc. (Lond.). Birmingham, Leicester, and Leamington: The Midland Educational Company, Lim. Pp. 31.

ALL the problems recently set at the Oxford and Cambridge Local College of Preceptors, London Matriculation, and South Kensington (Stages I. and II.) Examinations, will be found in this pamphlet, together with the answers and solutions. It will no doubt be useful to students who are about to present themselves for examination before any of these bodies, as it will give them a good idea of the kind of questions asked, and also the way they should shape their answers. It is essentially a book for beginners, and handy in form.

CORRESPONDENCE.

THE ATOMIC WEIGHT OF BORON.

To the Editor of the Chemical News.

SIR,—The perusal of Mr. F. P. Armitage's paper on "The Atomic Weight of Boron" (*CHEMICAL NEWS*, vol., lxxvii., p. 78) leads me to believe that some observations of my own may be of interest.

Some time ago I made a number of experiments on the behaviour of borax on ignition, paying especial attention to the loss of weight sustained at various temperatures and to the alkalinity of the residue. I came to the following conclusions:—

1. The loss of weight depends greatly upon the temperature and time of ignition. In one experiment, for example, the loss after a few minutes' ignition over a Bunsen flame amounted to 47.04 per cent, and after several hours' ignition to 47.37 per cent. In several experiments in which the blowpipe was used, a loss of over 50 per cent was noted, and when a closed crucible was used a sublimate was obtained on the lid.

2. The loss, beyond that accounted for by the water of crystallisation, is due to volatilisation of soda and of

boric acid. This is shown by the decrease in the amount of standard acid required to neutralise the residue.

3. It appears that the residue contains rather less soda than that required for the formula $\text{Na}_2\text{B}_4\text{O}_7$, and that the composition of the residue is not constant.

The use of borax for the determination of the atomic weight of boron seems, therefore, liable to be attended by two sources of error, due respectively to the uncertainty as to the composition of the hydrated and of the fused salt. It occurred to me that more trustworthy results might possibly be obtained by a series of experiments in which the alkalinity of the crystallised borax and that of the fused salt are compared together. So long as the acid required to neutralise the fused salt is not less than that required by the weight of crystalline borax from which it was obtained, it is clear that no loss of soda has been caused by ignition; and by taking into account also the loss of weight on ignition, it may be possible to select those experiments in which all water has been driven off and yet no decomposition has taken place from over-ignition.

I intend to make some experiments in this direction.—I am, &c.,

NORMAN LEONARD.

Chemical Laboratory, Guy's Hospital,
London, S.E., February 21, 1898.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Zeitschrift für Anorganische Chemie,
January 18, 1898.

Use of the Metals Sodium, Magnesium, and Aluminium in Qualitative Analysis.—Walther Hempel.—A small piece of sodium is flattened on filter-paper; this is sprinkled with the powdered substance which is to be analysed. The paper is rolled into a cylinder, and wound round with iron wire; it is then placed in the reducing flame of a Bunsen burner. The reaction is finished in a few seconds. On cooling, the spiral is unwound and the contents placed in an agate mortar. A little water dissolves the sodium salts, and the reduced metals are generally found in large enough quantities to be estimated by the ordinary methods. Sodium is not suitable where spectrum analysis is to be used; in this case magnesium or aluminium can be substituted for sodium; the method is the same, except that the powdered metal is mixed with the substance to be examined.

Quantitative Separation of Ethylene and Benzene Vapour.—E. Harbeck and G. Lunge.—In order to separate ethylene and benzene vapour in illuminating gas, first the oxygen and acetylene must be removed, and can be estimated; then the mixed gases are passed with hydrogen over platinum-black. The apparatus used is described, and a diagram given. By this process the ethylene is converted by the hydrogen into ethane, the benzene remaining unaltered. The contraction is measured. This reaction is, however, completely stopped if there is any carbon monoxide present in the mixture. Benzene can also be estimated as dinitrobenzene. When acted upon by concentrated nitric acid, or a mixture of this acid and concentrated sulphuric, it is nitrated to mononitrobenzene; but by bringing a very little benzene in contact with a large excess of acid the nitration goes further, and dinitrobenzene is formed. This is not very soluble in cold water, but easily soluble in alcohol and ether, and so can readily be separated out and estimated. At ordinary temperatures it is quite solid, and, though

somewhat yellow, even with the microscope drops of mononitrobenzene cannot be distinguished.

Action of Carbon Monoxide on Platinum and Palladium.—E. Harbeck and G. Lunge.—When ethylene is led over Pt or Pd, together with hydrogen at ordinary temperatures (18° — 100°), a contraction takes place, expressed by the equation $\text{C}_2\text{H}_4 + \text{H}_2 = \text{C}_2\text{H}_6$. The presence of carbon monoxide completely hinders this reaction, and so prevents the separation of ethylene from benzene vapour, by the method described in the former paper. This fact is explained as follows:—Both platinum and palladium form, with carbon monoxide, a compound which is stable enough to be called a true chemical compound (even though the whole of the platinum and palladium cannot be combined in a state pure enough for analysis), for the following reasons:—(1) It cannot be displaced by another gas, and even after treatment with hydrogen it will take up the same quantity of carbon monoxide as if the hydrogen were not there. (2) If the compound of carbon monoxide and platinum is heated to 250° , the carbon monoxide is given off suddenly. (3) Carbon monoxide forming a compound prevents the free surface of the platinum coming in contact with the ethylene and hydrogen, and thus stops their reaction.

Note on the Methods of Estimating the Carbon in Iron.—E. Harbeck and G. Lunge.—The amount of carbon in cast-iron and steel was estimated by several methods, the following conclusions being arrived at:—(1) The chlorine method and Corleis's method give results which agree well. (2) The method of Lunge and Marchlewski gives worse results than Corleis's method, except in the case of steel, when the copper sulphate is replaced by copper-ammonium chloride. In the case of cast-iron the gain in accuracy by using copper-ammonium chloride is so little that it is of no value. (3) The copper-ammonium chloride method requires longer time, and is much more detailed than the method of Lunge and Marchlewski, and the results of the analysis are very slightly more accurate. (4) For an accurate analysis Corleis's method is recommended, but for commercial use the method of Lunge and Marchlewski; the latter can be worked with rapidity and ease. A constant loss of about 0.03 per cent must be allowed for.

Manganese Molybdates.—Arthur Rosenheim and Hermann Itzig.—(1). Ammonium manganese molybdate was prepared by Péchard's method and analysed. The manganese was found to be present as sesquioxide, the formula being $2(\text{NH}_4)_2\text{OK}_2\text{OMn}_2\text{O}_3 \cdot 10\text{MoO}_3 + 5\text{H}_2\text{O}$. (2). The potassium manganese molybdate was prepared by three different methods, and in each case appeared to have the formula $3\text{K}_2\text{OMn}_2\text{O}_3 \cdot 8\text{MoO}_3 + 5\text{H}_2\text{O}$. The difference in the results obtained by this method and Péchard's has not been properly explained, as, in spite of many experiments, no salt of a different composition from the above formula could be obtained. The change of composition took place not only in the direct preparation, but when the ammonium potassium double salt changed into the potassium salt.

Journal de Pharmacie et de Chimie,
Series 6, vol. vii., No. 2.

Transformation of Sorbite into Sorbose by Mycoderma vini.—A paper describing a number of experiments on fermentation of the juices extracted from the berries of the *Sorbus aucuparia*; not suitable for abstraction.

A New Water Ureometer.—A. Chassevant.—This ureometer is composed of a tube graduated to tenths of a c.c., surmounted by two blown bulbs, themselves united by means of a lateral syphon, which practically makes the upper bulb a Tantalus cup. The interior arrangement of the tubes allows the gas to circulate freely through the graduated tube and the two bulbs, while preventing the liquid contained in the bulbs from entering

the graduated tube. A full description of the method of procedure for making an estimation is given, and it is claimed that—besides its application to the estimation of urea—this apparatus can be used for estimating the total nitrogen by Henninger's method, and also for making exact measurements of the volumes of gases set free by the action of one liquid on another.

Rotatory Power of Chlorhydrate of Cocaine.—H. Herissey.—These experiments have been carried out on five totally different samples, and they have given very concordant results, three of them having an identical rotatory power, viz., 71.66° in an aqueous solution of 2 grms. per 100 c.c.; under the same circumstances the rotatory power of the other two was 70.83° . At first sight these figures appear to be high, $\alpha_D = -52.5^\circ$ being the value given in the "Codex" and in other works. The author has carefully examined and repeated the work done by Antrick on this subject, and comes to the certain conclusion that the results obtained by him were wrongly interpreted, and that about 71.66° is the correct figure. It is possible, he admits, that the material used by Antrick in 1887 may have been of less purity than it can be obtained at the present time, and, further, he remarks that he has never experienced any cloudiness in his aqueous solutions of chlorhydrate of cocaine, an occurrence which obliged his predecessor in this matter to make use of dilute alcohol.

Series 6, vol. vii., No. 3.

On a New Ferment from the Tartrates, "Bacillus tartricus."—L. Grimbert and L. Ficquet.—An anaerobic fermentation of tartrate of lime, induced by means of a few drops of a vegetable juice, incubated at 35° , was the starting-point of this research. After a number of cultivations the authors were enabled to isolate a new bacillus, to which they have given the name *Bacillus tartricus*. It is a small bacillus, about 1 to 2 μ in length, with considerable motile power; it gives no indol reaction in a peptone solution; it coagulates milk about the eighth day, and transforms nitrates into nitrites. *B. tartricus* is an active ferment of tartrate of lime, which it attacks differently in aerobic or anaerobic cultures, but shows a preference for aerobic life.

Use of Carbide of Calcium for the Preparation of Absolute Alcohol.—P. Yvon.—This paper has already been inserted in full.

Some New Derivatives of Pyrocatechin.—H. Cousin.—The author has succeeded in obtaining trichloridised pyrocatechin, by acting on an acetic solution of pyrocatechin with an acetic solution of chlorine in the proportion of 6 atoms of the former to 1 molecule of the latter. He has also prepared two dibromised pyrocatechins as well as several mononitrated derivatives. By the action of sulphuric acid of different strengths, he obtained a monosulphuric pyrocatechin and a disulphonic pyrocatechin.

MISCELLANEOUS.

The British Fire Prevention Committee.—Mr. Robert Mond, M.A., F.R.S.E., has joined the working Executive of this Committee, with the particular view to attending to the interests of the chemical side of Fire Prevention and the questions of research work.

International Congress of Hygiene and Demography.—Referring to our recent notice of this Congress and Exhibition, to be held at Madrid early in April next, we are informed that full details can be obtained from Sir Douglas Galton, F.R.S., Parkes Museum, 74 a, Margaret Street, London, who is the President of the National Committee.

New Method for the Estimation of Casein in Milks.—G. Deniges.—Twenty-five c.c. of milk is placed in a 200 c.c. matrass; to this is added 5 c.c. of a cold saturated solution of oxalate of ammonium, 20 c.c. of a N/10 solution of mercuric-potassic iodide, 2 c.c. of acetic acid, and the whole made up to 200 c.c. with distilled water, and filtered. 100 c.c. of this filtrate is taken, and 10 c.c. of a solution of cyanide of potassium (equivalent to the decinormal argentic solution) is added, then 12 to 15 c.c. of ammonia. Add to this, drop by drop, N/10 nitrate of silver until the solution is permanently clouded (g). In another vessel place 10 c.c. of the same cyanide solution, 12 to 15 c.c. of ammonia, 10 c.c. of N/10 mercuric-potassic iodide, and 100 c.c. of water. Run in the N/10 solution of nitrate of silver until the cloudiness remains (c); (g-c) corresponds to the casein in the milk under examination. In case g-c is higher than 37, it is advisable to dilute the milk by one-half, as the operation is much more sensitive with dilute than with concentrated solutions of casein.—*Journal de Pharmacie et de Chimie*, Series 6, vol. vii., No. 1.

MEETINGS FOR THE WEEK.

- MONDAY, 7th.—Society of Arts, 8. (Cantor Lectures). "The Principles of Design in Form," by Hugh Stannus, F.R.I.B.A.
— Royal Institution, 5. General Monthly Meeting.
- TUESDAY, 8th.—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
— Society of Arts, 8. "The Making of a Stained Glass Window," by Lewis Foreman Day.
- WEDNESDAY, 9th.—Society of Arts, 8. "Linde's Method of Producing Extreme Cold and Liquefying Air," by Prof. J. Ewing, F.R.S.
- THURSDAY, 10th.—Society of Arts, 4.30. "India and Sir Henry Maine," by Charles Lewis Tupper, C.S.I.
— Royal Institution, 3. "Recent Researches in Magnetism and Diamagnetism," by Prof. J. A. Fleming, M.A., D.Sc., F.R.S.
- FRIDAY, 11th.—Physical, 5. "On Dynamical Illustrations of certain Optical Phenomena," by Prof. J. D. Everett, F.R.S.
— "On Properties of Liquid Mixtures," by R. A. Leffeldt.
— Royal Institution, 9. "Marked Unexplored," by Walter Frewen Lord.
- SATURDAY, 12th.—Royal Institution, 3. "English Letter Writers," by Professor Walter Raleigh, M.A.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Manufacture of Glucose-sugar.—Can anyone give me the name and publisher of a practical chemical work on the modern manufacture of glucose-sugar? I find considerable difficulty in neutralising the sulphuric acid; chalk seems to leave a considerable quantity of free acid in the sugar.—S. CHIVERS.

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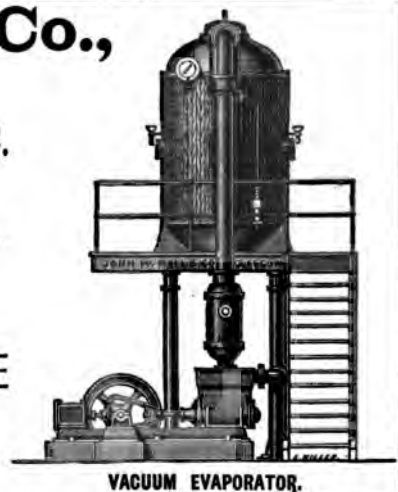
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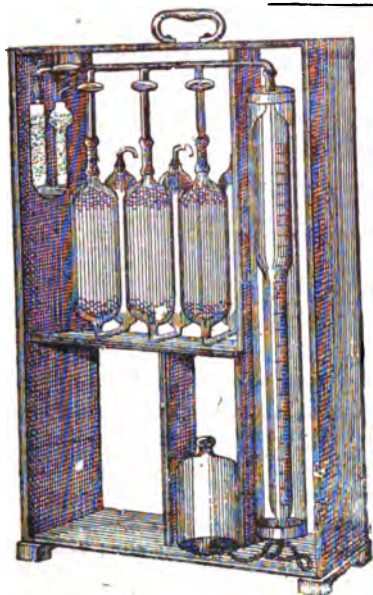
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CONTENTS.

ARTICLES:—	PAGE
A Quick Polarimetric Method for the Estimation of Starch in Flour, &c., by Edwin Dowdard	107
On the Rapid Detection and Estimation of Manganese in Plants and Vegetable Soils by a Colorimetric Method, by P. Pichard	108
The Analysis of Sulphide and Sulphydrate of Sodium, by P. Dobriner and W. Schranz	108
The Determination of the Carbonic Acid in Bicarbonates, by G. Lunge	109
On the Effect of Light on the Combination of Hydrogen and Bromine at High temperatures, by J. H. Kastle and W. A. Beatty	111
A Delicate Test for the Detection of a Yellow Azo Dye used for the Artificial Colouring of Fats, &c., by Joseph F. Geisler	112
Liquid Air by the Gallon	115
PROCEEDINGS OF SOCIETIES:—	
CHEMICAL SOCIETY.—A Chemical Examination of the Constituents of Indian and American Podophyllum—The Volatile Constituents of the Wood of <i>Goupia tomentosa</i> —&c.	113
ROYAL INSTITUTION	115
CHEMICAL NOTICES FROM FOREIGN SOURCES	116
MISCELLANEOUS	117
MEETINGS FOR THE WEEK	118
TO CORRESPONDENTS	118

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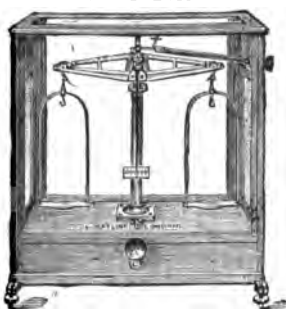
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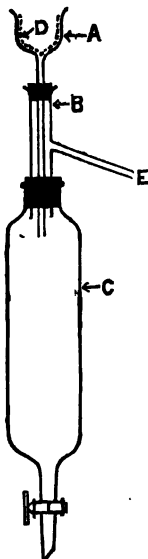
A QUICK POLARIMETRIC METHOD FOR THE ESTIMATION OF STARCH IN FLOUR, &c.

By EDWIN DOWZARD.

This method depends upon the conversion of starch into optically active bodies by the agency of diastase.

A good malt extract is obtained, such as Kepler's, or that manufactured by the Britannia Malt Extract Company; any extract, however, will do if 1 grm. of it is capable of transforming an equal weight of potato starch (as mucilage) into dextrin and maltose in the space of twenty minutes, the mixture being kept at 40-5° C. Fifty grms. of this extract is dissolved in 500 c.c. of cold water, 5 grms. of washed kaolin added, the mixture agitated, and poured on a large double filter, only that portion which passes through clear being collected.

One grm. of flour is mixed with a small quantity of cold water, 35 c.c. of boiling water is added, the mucilage kept at 100° C. for thirty seconds, then cooled down to 48° C. and treated with 20 c.c. (accurately measured) of the above malt extract solution; the mixture is maintained at 48° C. for twenty minutes, and then just brought to the boiling-point and filtered. The following piece of apparatus will be found useful at this point, as the filtration is slow.



- A. Thistle-headed funnel.
- B. Top portion of a fractional distillation flask.
- C. Cylindrical separator.
- A small circular piece of lint, D (represented by dotted lines), is placed in cup of funnel.
- E is attached to a filter-pump.

The filtrate is cooled and made up to 100 c.c., a small quantity of kaolin is added, and the whole poured on a dry double filter; the optical rotation of the clear filtrate is then taken in a 20 c.m. tube, using an instrument of the Laurent half-shadow type. (It is more convenient to take the sugar-divisions than the angular-degrees—100 sugar divisions = +21° 40').

Twenty c.c. (accurately measured) of the malt extract solution is diluted with 50 c.c. of water, boiled, then cooled and made up to 100 c.c.; the whole is filtered, the optical rotation of the filtrate being taken as before.

The difference between the two results is due to the starch and a small quantity of dextrine contained in the flour.

As flour contains variable quantities of dextrine it will be necessary to apply a correction which is obtained in the following manner:—One grm. of flour is converted into mucilage as before and made up to 100 c.c. with water, 1 grm. of kaolin is added, the mixture raised to 100° C., and poured on a large, dry, double filter; it is only necessary to filter enough to fill a 20 c.m. tube, in which the optical rotation is taken; this, after being multiplied by 2, is added to the malt extract figure before subtraction.

One grm. of starch in 100 c.c. = +14.4 sugar divisions (+3° 7').

The following experiments were made with pure (wheat and potato) starch, dried *in vacuo*, and show that the above method is trustworthy.

In the first series the diastase was allowed to act for twenty minutes at 48° C. before bringing it to the boiling-point; in the second, three hours were allowed to elapse before boiling; different malt extracts were used in the two series.

The results show that after twenty minutes the optical activity is constant.

Wheat Starch. Twenty minutes at 48° C.

	Sugar divisions.		Sugar divisions.
1 grm. starch + 20 c.c. malt solution	+26.1	2 grms. starch + 20 c.c. malt solution	+40.5
20 cc. malt solution		20 c.c. malt solution	
— starch	+11.7	— starch	+11.7
	+14.4		+28.8
1 grm. wheat starch in 100 c.c.	+14.4	2 grms. wheat starch in 100 c.c.	+28.8

Potato Starch. Twenty minutes at 48° C.

	Sugar divisions.		Sugar divisions.
1 grm. starch + 20 c.c. malt solution	+26.1	2 grms. starch + 20 c.c. malt solution	+40.5
20 c.c. malt solution		20 c.c. malt solution	
— starch	+11.7	— starch	+11.7
	+14.4		+28.8
1 grm. potato starch in 100 c.c.	+14.4	2 grms. potato starch in 100 c.c.	+28.8

Wheat Starch. Three hours at 48° C.

	Sugar divisions.		Sugar divisions.
1 grm. starch + 20 c.c. malt solution	+26.7	2 grms. starch + 20 c.c. malt solution	+41.1
20 c.c. malt solution		20 c.c. malt solution	
— starch	+12.3	— starch	+12.3
	+14.4		+28.8
1 grm. wheat starch in 100 c.c.	+14.4	2 grms. wheat starch in 100 c.c.	+28.8

Potato starch gave exactly the same results as wheat starch (after being kept for three hours at 48° C.).

(The optical rotation in all cases is taken at 16° C.).

A very simple calculation enables us to arrive at the percentage of starch in the sample operated upon. For instance—

	Sugar divisions.
1 grm. flour + 20 c.c. malt solution ..	+22.7
	+13.3
	+9.4
20 c.c. malt sol. minus flour =	+12.3
Correction for dextrin, &c. =	+ 1.0
	13.3
14.4 : 9.4 :: 100 : x	
= 65.2	

The flour therefore contains 65.2 per cent starch.

Once the malt extract solution is prepared, an estimation of starch in flour, &c., can be completed within two hours.

ON THE RAPID DETECTION AND ESTIMATION OF MANGANESE IN PLANTS AND VEGETABLE SOILS, BY A COLORIMETRIC METHOD.

By P. PICHARD.

WHEN the soil contains any notable quantity of manganese, fusion with an alkaline carbonate gives a characteristic green colouration, which can be confirmed by the rose-colour produced by the addition of nitric acid.

Test for Manganese in Vegetable Soils.—The soil is dried and finely pulverised, then incinerated at a dull red-heat in a small platinum crucible, after which it is calcined with dried and powdered carbonate of soda or potash. We take from 0.5 to 1 grm. of the soil according as the colouration is more or less pronounced after the incineration; the residue is then thoroughly mixed with two parts of the alkaline carbonate. The vitreous or opaque mass produced is detached by adding a little water acidulated with nitric acid; this is placed in a test-tube, and about 0.5 grm. of minium or of binoxide of lead is added. Four c.c. of water and 2 c.c. of pure nitric acid are then poured in. Heat to boiling until the volume of the liquid is reduced by one-half; let stand until the insoluble matter has settled; the supernatant liquid should be rose-coloured if the earth contains traces of manganese.

Test for Manganese in Organic Matters.—The organic matter is dried, then rubbed fine in a mortar, incinerated, calcined with an alkaline carbonate, and treated in the manner which has been described for a vegetable soil. The incineration, however, should be carried out at a slightly higher temperature. A few decigrams. of the ash will be enough for the test. The calcination with the alkaline carbonate should be effected at a bright red-heat.

Estimation of the Manganese.—The principle consists in transforming the manganese into a permanganate dissolving to a solution of a rose-colour, and in comparing the tint of this solution with that of a typical solution containing a known quantity of manganese. The solutions are placed in graduated test-tubes of the same diameter, and distilled water is added until the colours correspond.—*Comptes Rendus*, cxvii., No. 7.

THE ANALYSIS OF SULPHIDE AND SULPHYDRATE OF SODIUM.

By P. DOBRINER and W. SCHRANZ.

SOME little time ago we had occasion to analyse a product offered for sale under the name of "sulphydrate of sodium." Analysis showed that it was nothing more than ordinary sulphide of sodium. We have also often

had to estimate the proportion of free alkali in commercial sulphide of sodium. We think it would be of use to publish the method we employ for this class of analysis, which is characterised by great simplicity.

As is well known, the sulphydrate NaSH is produced by the action of sulphuretted hydrogen on sulphide of sodium, $(\text{Na}_2\text{S} + \text{gH}_2\text{O}) + \text{H}_2\text{S} = 2\text{NaSH} + \text{gH}_2\text{O}$. On the other hand, by adding the theoretical quantity of caustic soda to sulphydrate of sodium dissolved in water, sulphide of sodium is formed, $\text{NaSH} + \text{NaOH} = \text{Na}_2\text{S} + \text{H}_2\text{O}$. It thus results that the co-existence of sulphide of sodium, of sulphydrate of sodium, and of caustic soda in one solution is *a priori* impossible. Analysis can, therefore, only have for its object the estimation of sulphide of sodium side by side with sulphydrate of sodium, and the estimation of sulphide of sodium side by side with caustic soda.

I.—Estimation of Sulphide of Sodium in the presence of Sulphydrate of Sodium.

Dissolve 12 grms. of the substance to be analysed in 1 litre of water, and allow about 25 c.c. of this solution to run, by means of a burette, into 45 c.c. of a $\frac{1}{10}$ normal solution of iodine, previously acidulated with 10 c.c. of a normal solution of sulphuric acid, and diluted with water to form 180 c.c.

The yellow colour of the solution should disappear. If the contrary is the case, the experiment must be repeated with a smaller proportion of the $\frac{1}{10}$ normal solution of iodine. After adding a little starch solution, we estimate by iodine solution the excess of sulphuretted hydrogen set at liberty. By multiplying the number of c.c. of iodine solution employed by 2, we obtain the percentage of sulphide of sodium present in the substance:—Let *a* be the percentage of $(\text{Na}_2\text{S} + \text{gH}_2\text{O})$ found, then the total quantity of sulphuretted hydrogen obtained from the substance will be $\frac{34}{240}a$ per cent H_2S .

On the other hand, we dissolve 6 grms. of the substance in water, treat the solution with an excess of normal sulphuric acid, boil to drive off the sulphuretted hydrogen, and titrate the excess of acidity with a normal solution of caustic soda, using phenolphthalein as indicator. The difference between the total acidity and the acidity remaining multiplied by 2, again gives the percentage of sulphide of sodium present. The figure obtained corresponds with the sulphuretted hydrogen combined with the sodium. Let *b* be the percentage of $(\text{Na}_2\text{S} + \text{gH}_2\text{O})$ found in the second operation, then the quantity of combined sulphuretted hydrogen will be $\frac{34}{240}b$ per cent H_2S .

The mixture of sulphide of sodium and sulphydrate of sodium may be considered as a mixture of sulphide of sodium and free sulphuretted hydrogen. We have found that the total amount of sulphuretted hydrogen = $\frac{34}{240}a$ per cent, and that the sulphuretted hydrogen combined with the sodium was = $\frac{34}{240}b$ per cent; the quantity of free sulphuretted hydrogen present is therefore = $\frac{34}{240}(a-b)$ per cent H_2S .

The quantity of sulphide of sodium is = *b* per cent. According to the equation—



the $\frac{34}{240}(a-b)$ per cent ought to combine as follows—

$$\frac{240}{34} \times \frac{34}{240}(a-b) = a-b \text{ per cent } (\text{Na}_2\text{S} + \text{gH}_2\text{O})$$

to form—

$$\frac{112}{34} \times \frac{34}{240}(a-b) = \frac{7}{15}(a-b) \text{ per cent NaSH.}$$

The substance examined, therefore, contained—

$$b - (a - b) = 2b - a \text{ per cent } (\text{Na}_2\text{S} + 9\text{H}_2\text{O})$$

and—

$$\frac{7}{15} (a - b) \text{ per cent NaSH.}$$

II.—Estimation of Sulphide of Sodium in presence of Caustic Soda.

We determine the total amount of sulphuretted hydrogen by means of the iodine solution, and express the result in terms of sulphide of sodium.

Let x be the per cent of $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$ found. The quantity of caustic soda combined with the sulphuretted hydrogen is then $\frac{80}{240} x$ per cent NaOH.

We determine by titration the total amount of free caustic soda and the soda combined with the sulphuretted hydrogen, and similarly express the result in terms of sulphide of sodium. The amount found = y per cent $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$. The amount of free and combined sodium is, therefore, $\frac{80}{240} y$ per cent. The difference between these figures represents the amount of free soda present:—

$$\frac{80}{240} (y - x) = \frac{1}{3} (y - x) \text{ per cent.}$$

Thus, the substance contains x per cent of $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$ and $\frac{1}{3} (y - x)$ per cent of free NaOH.

It results from the preceding, that the substance examined contains sulphhydrate when the proportion of $\text{Na}_2\text{S} + 9\text{H}_2\text{O}$ present (found by means of the iodine solution) is higher than that found by titration with normal acid and soda. In the contrary case the substance contains free alkali.

By this method, no account of the possible presence of polysulphides, sulphites, &c., is taken. The presence of these impurities would, without doubt, complicate the analysis; but in most cases they are neglected in the analysis of sulphide of sodium.—*Zeit. für Angewandte Chemie*, p. 455, 1897.

THE DETERMINATION OF THE CARBONIC ACID IN BICARBONATES.

By G. LUNGE.

It is well known that the carbonic acid in the bicarbonates can be estimated in several manners with more or less exactness. I myself have already described three methods at different times:—(1) The method by ammonia and chloride of barium (the method by using soda and chloride of barium gives almost identical results); (2) since then, as the most exact method, the estimation in the gaseous state of the total carbonic acid. These two methods are described in the "*Vade mecum* of the Manufacturer of Chemical Products," by G. Lunge, p. 223; (3) a very short time since, the method of Sundström. According to this last, we add a solution of soda and baryta to the solution of the bicarbonate until one drop of the liquid gives an immediate brown precipitate with nitrate of silver by the touch test (*Zeit. für Angew. Chem.*, 1897, p. 169).

In order to determine the bicarbonate by either of these three methods, it is also necessary to know the alkali-metric titration of the bicarbonate. The third of these methods leads easily to this, but it requires a previous assay of a bicarbonate of known strength. If this precaution be neglected, one may be—as I well know—led into considerable errors. Further, this method is very useful as a control in manufacture, &c., though it is not sufficiently certain for exact determinations.

I have at last, however, succeeded in finding a means for the determination of the carbonic acid in bicarbonates

—applicable only, it is true, to solid bodies, but which possesses all the necessary qualities. The operations are very simple and rapid, and give results of as great accuracy as could be desired. In this manner my new process far exceeds all the others.

This new method is of surprising simplicity; it consists only of heating the material to be analysed, and estimating the volume of the carbonic acid set free. The reasons why such a method has not hitherto been thought of are diverse. The first is that it is impossible to completely set free the carbonic acid of the bicarbonates when working with an alkaline solution. I have quite recently re-confirmed this opinion after a large number of experiments, in which I used vacua and modes of procedure of the most varying kind. For solutions it is necessary, as before, to use the old methods; for bodies in the solid state, a special apparatus is further required for the successful application of the new method. I first attempted to utilise the carbonic acid apparatus described by M. Marchlewsky and myself (*Zeit. für Ang. Chemie*, p. 229, 1897). After having placed the substance in the small disengagement chamber, the air was exhausted as thoroughly as possible; the whole was then heated in an air-bath. The carbonic acid was estimated by absorption; what remained in the apparatus was drawn out by a current of air and measured afresh; this last operation was repeated three or four times.

By this rather complicated operation, I obtained results varying from 91 to 101 per cent of the real quantity present. I cannot exactly explain the cause of these variations, in every case there are several sources of error of which I will not speak at the present time, since we cannot achieve what is required.

I have myself been astonished at the results arrived at by the apparatus now used; it resembles one of my "bulb nitrometers," or perhaps, to be more correct, one of my "gas-volumeters"; and, further, it can be made to communicate with any convenient apparatus for measuring or weighing the carbonic acid, taking care always that it has as little waste space as the "gas-volumeter"; this is always difficult to obtain with apparatus for gravimetric estimations.

A glass tube 65 m.m. long and 6 m.m. internal diameter is terminated at one end by a short length of tube of greater diameter, and plugged with a ground glass stopper; at the other end is a capillary tube 60 m.m. in length. The plug is attached to a glass rod 30 m.m. in length, unground, but of a diameter almost equal to the internal diameter of the tube; there thus remains a free space of 35 m.m. in length and of 6 m.m. in diameter, which is plugged at the capillary end by a little asbestos or glass wool. The cavity will hold about 0.850 grm. of powdered bicarbonate of soda; that is to say, an amount which, with a substance of good quality, will give a little more than 110 c.c. of carbonic acid at 0° and 760 m.m. pressure, a very convenient quantity for the universal "gas-volumeter," the graduations of which are from 1 to 30 c.c. and from 100 to 140 c.c., while the space between 30 and 100 c.c. is occupied by a bulb. If this cavity is too small or too large for any particular estimation, it can easily be altered by cutting off part of the prolongation of the plug in the first case, or by introducing more glass wool or asbestos in the second case.

The prolonged unground end of the plug is to allow of the heating of the cavity without incurring the danger of the ground part cracking, the latter, as is well known, being very sensitive to variations of temperature; we can also, without inconvenience, smear the ground part outside the air-bath with vaseline or fat, so as to make the joint perfectly tight. The heating is done *in situ* by means of an air-bath formed of a small iron crucible having two holes diametrically opposed to one another; this is covered by a sheet of asbestos through which a thermometer passes. Another sheet of asbestos is placed over the capillary tube, and this reaches to below the flame of the burner used for heating the crucible, and protects the

"gas-volumeter" from lateral heating. But in spite of this precaution it is necessary to wait at least ten minutes after each experiment before levelling and taking readings.

The operation is one of extreme simplicity. The exhausted tube is weighed, the bicarbonate introduced, and any adhering to the part near the plug carefully wiped away; the latter is adjusted with a little vaseline and the whole weighed afresh. The tube is then placed in the air-bath, and the capillary tube is connected with the lateral capillary tube of the "gas-volumeter." A vacuum is then made by lowering the mercury reservoir two or three times until a barometric vacuum is formed in the measuring tube, closing the connection on the side of the capillary tube and driving out the air; this operation should be repeated at the rate of about three times a minute.

The reservoir is again lowered, and the air-bath heated by a moderate sized flame until the thermometer indicates 260° to 270° ; this will take about ten minutes. This temperature must be maintained for another three minutes, when the tap of the measuring tube must be closed. The apparatus is then allowed to cool for ten minutes, and the reservoir and reduction tube arranged so that the temperature and pressure are reduced to 0° and 760 m.m., when the volume of gas can be read off. The reduction tube should be arranged for moist gas, as the gas given off is always saturated with water. The condensation of water is, however, always so slight that it cannot have any influence on the reading. We can calculate the carbonic acid from the bicarbonate from the volume obtained, for, after having made a vacuum three times, the empty space no longer contains any measurable quantity of air. I have, however, always preferred after each reading to pass the gas into an Orsat tube, so as to be quite certain that it was completely absorbed by potash, which, I may add, always occurred. One c.c. of carbonic acid gas at 0° and 760 m.m. weighs, as is well known, 1.96633 m.grms., and this corresponds to 7.5124 m.grms. of NaHCO_3 ($O = 16$, $H = 1.008$, $C = 12.01$, $Na = 23.05$). We multiply the number of c.c. found by this figure, divide by the weight of the substance used, and thus obtain the proportion of bicarbonate.

If we wish to know the proportion of normal carbonate we determine the alkalimetric titration in another experiment (by means of normal acid and methyl-orange), or else the total carbonic acid by decomposition by means of hydrochloric acid, according to the method described by Marchlewski and myself.

For the purpose of controlling this new method, we first analysed a commercial bicarbonate of good quality by the old method.

By titrating with one-fifth normal hydrochloric acid, I obtained its alkalimetric value calculated into Na_2CO_3 : 63.09—63.10—63.11—63.11, or a mean of 63.10 per cent (b).

Total carbonic acid:—

51.91 p. c. CO_2 corresponding to	125.16 Na_2CO_3
51.87 " "	125.12 "
51.98 " "	125.34 "
Mean 51.92 "	125.21 (a),

According to the formula—

$$\frac{168.136}{106.11} (b-a) = 1.5845 (b-a),$$

we obtain a proportion of bicarbonate of 98.41 per cent. The possible error of 0.1 per cent in the determination of the alkalimetric value, or of the total carbonic acid (which may easily happen), would have a great influence on the proportion of bicarbonate present; in the first case the difference would be 0.16 per cent, and in the second 0.40 per cent. We thus see of what great importance the estimation of the carbonic acid is; we can obtain it

with the necessary degree of accuracy by the "gas-volumeter" method.

The direct determination of the bicarbonate by the apparatus described above gives the following results:—

Substance used.	CO_2 , 0° , 760 m.m. C.c.	CO_2 , M.grms.	CO_2 , Per cent.	NaHCO_3 , Per cent.
841.0	110.4	217.08	25.81	98.62
843.8	110.9	218.06	25.84	98.73
847.9	111.5	219.25	25.86	98.79
852.8	112.1	220.43	25.84	98.73
846.5	111.3	218.85	25.85	98.77
843.6	110.9	218.06	25.85	98.75
870.0	114.4	224.95	25.86	98.79

The mean of all these determinations is 98.74 per cent of NaHCO_3 . If we reject the first, as it is rather too far from the mean of the others (which may reasonably be allowed in a first determination), the average becomes 98.76 per cent, and no single determination varies by more than 0.03 per cent. It must be admitted that this is one of the most exact methods of chemical analysis, and it is so much the more certain as it gives the substance sought for directly—that is, the carbonic acid in the state of bicarbonate. The result is much more satisfactory than that of the first one, which showed a deficit of 0.35 per cent NaHCO_3 ; it would make one think that an error of +0.05 per cent in the titration, and of -0.05 per cent in the determination of the total carbonic acid, would cover all differences, and no one can guarantee results to within +0.05 per cent. This, again, shows the great advantage of the new direct method over the old indirect method.

If we further want to know the amount of normal carbonate of soda contained in the bicarbonate, we take the alkalimetric value of the substance (which in the present case had already been done), calculate the titration value in Na_2CO_3 (= 63.10 per cent), and subtract the equivalent of the bicarbonate:—

$$\frac{98.76 \times 53.065}{84.07} = 62.33;$$

$63.10 - 62.33 = 0.77$ per cent, which is the figure sought for.

Further, to avoid an extra weighing, we might dissolve the residue contained in the small tube, and titrate it; but we might experience a small loss more easily than by weighing out a fresh quantity. As a rule, however, it is not necessary to carry out this operation.

We can also find the normal carbonate by estimating the total carbonic acid and deducting double the quantity of carbonic acid in the bicarbonate, &c. In the present case we should have $51.92 - (2 \times 25.84) = 0.24$ per cent $\text{CO}_2 = 0.58$ per cent of normal carbonate. Is this result more accurate than the preceding (0.77 per cent)? I would not care to confirm it; we can never expect great exactitude in analyses by difference.

I wish particularly to draw attention to the fact that the complete displacement of the carbonic acid in the bicarbonates, which I have just described, does not require the carbonate of soda destined for the titration of the normal acids to be heated above 300° ; it is preferable to heat it on a sand- or air-bath. We can be sure, if this operation is sufficiently prolonged (for a few grms. half an hour to an hour suffices), that there is no more bicarbonate remaining, and that no oxide of sodium is yet formed, as might happen if the temperature reached a dull red-heat.

In conclusion, I wish to say that M. Ernest Harbeck has been of very great assistance to me in this work.

The necessary tubes can be easily made by any glass-blower; they can be obtained, however, from M. Stadelmann, Neumarkt, Zurich, together with the crucible and the air-bath, for two francs.—*Zeitschrift für Angewandte Chemie*, p. 522.

ON THE
EFFECT OF LIGHT ON THE COMBINATION OF
HYDROGEN AND BROMINE AT HIGH
TEMPERATURES.

By J. H. KASTLE and W. A. SEATTY.

THE combination of hydrogen and chlorine to form hydrochloric acid has been, and is still, the subject of a great deal of investigation. The effect of light, especially on this combination, has been very thoroughly studied by Draper, Bunsen and Roscoe, and others. In this connection it has been observed by Amato that a mixture of hydrogen and chlorine, if cooled to -12°C ., could be exposed to direct sunlight for hours without combination taking place. Such being the case it occurred to one of us (Kastle) that sunlight ought to cause the combination of hydrogen and bromine, or at least accelerate the change of these elements into hydrobromic acid, if the temperature be raised sufficiently to enable them to combine at all. So far as could be discovered, no studies of this kind had ever been undertaken. It is stated, however, that hydrogen and bromine do not combine at ordinary temperatures even in the sunlight. In order, therefore, to determine the effect of light on this reaction, a number of glass bulbs were made out of a long piece of glass tubing, in such a way as to leave one bulb connected with another in series by means of a short glass tube of small bore. Hydrogen was then passed through the series of bulbs, and, after they had been filled with the gas, the hydrogen was allowed to bubble through a small quantity of bromine, which had been purified by distillation over

sodium bromide, and thus saturated with bromine it was passed through the bulbs. In this way the bulbs were filled with hydrogen more or less saturated with bromine vapour. The most remote necks of the end bulbs of the series were then sealed off in the flame of the blowpipe; and a series of bulbs thus obtained each of which contained very nearly, if not exactly, the same quantities of hydrogen and bromine. Each bulb was then sealed off in such a way as not to allow any of the gases to escape. The exact amount of hydrogen and bromine in the bulbs was not determined; it is known, however, that hydrogen was always present in excess. The hydrogen used in the experiments of series Nos. I. and II. was only purified by passing the gas through potassium permanganate and water, whereas the hydrogen employed in the experiments of series No. III. was very carefully purified by using a little copper sulphate in the sulphuric acid employed in making the gas, and by passing the gas through a solution of copper sulphate, then through one containing potassium permanganate, then through water and a solution of silver nitrate, and finally through pure water. Further, the hydrogen employed in all of the experiments herein described was not dried but used moist. Judging from certain preliminary experiments, it was thought best to work at temperatures in the neighbourhood of 200°C ., and *o*-toluidine boiling at 196°C . was selected as a suitable medium in which to heat the bulbs, the manner of conducting the experiments being about as follows:—

The bulbs containing the mixture of hydrogen and bromine were placed in large, thin-walled glass tubes, containing about 50 c.c. of *o*-toluidine, and so arranged as to be mainly in the vapour of the boiling toluidine and not in the liquid itself. The large glass tubes were closed

Series No. I. Temperature 196° .			
No. of Expt.	Time.	Condition.	Amount of change.
(1).	1 hour.	Sunlight.	Change nearly complete, only slight colour of bromine remaining.
(2).	2 hours.	Dark.	Very slight, if any, difference in colour of bromine. Very little change.
(3).	$\frac{1}{2}$ hour.	Sunlight.	Colour intermediate between (1) and (2). About half bromine combined.
Series No. II. Temperature 196° .			
No. of Expt.	Time.	Condition.	Amount of change.
(1).	95 minutes.	Sunlight.	Nearly complete.
(2).	5 hours.	Diffused light.	Colour intermediate between (1) and (3), this series. About half the bromine combined.
(3).	3 hours.	Dark.	Colour about as deep as in unexposed bulbs. Very little, if any, change.
Series No. III. Temperature 196° .			
No. of Expt.	Time.	Condition.	Amount of change.
(1).	15 minutes.	Sunlight (a).	Over 50 per cent of bromine combined.
(2). Same bulb as used in (1).	30 minutes.	Sunlight.	Almost complete—only traces of bromine left.
(3).	1 hour.	Dark.	No change observable.
(4). Same bulb as used in (3).	15 minutes.	Sunlight.	About 50 per cent change.
(5). Same bulb as used in (3) and (4).	15 minutes.	Sunlight.	Intermediate in colour between (4) and (6).
(6) b. Same bulb as used in (3), (4), and (5).	15 minutes.	Sunlight.	Change nearly complete—only traces of bromine remaining.

- (a) The bulbs used in the experiments of Series No. III. were the only ones exposed to strong sunlight. During the time of the experiments of Series I. and II. the sky was frequently overcast by thin white clouds, so that the exposures to the direct sunlight were frequently interrupted.
- (b) The bulb used in experiments 3, 4, 5, and 6 was opened and tested for hydrobromic acid. On opening the bulb dense white fumes appeared. A little water was run into the bulb and the solution thus obtained gave the well-known reactions of hydrochloric acid,

with corks bearing a long piece of glass tubing, to serve as a reflux condenser. Some of the bulbs, thus arranged, were heated in the dark and some in the sunlight. Only bulbs of the same series, of course, were compared as to the effect of light, and the amount of change was measured by the alteration in the colour of the bromine.

Appended are the results of three series of experiments. These results certainly go to prove that light causes the combination of hydrogen and bromine, at 196° C., the amount of change being proportional to the time of exposure to light. In the dark, at 196° C., the combination of these two gases is exceedingly slow, whereas, in the sunlight, the change is fairly rapid at this temperature. Further, certain results which have been obtained would seem to indicate that light causes the combination of these elements even at 100° C., although further experiments will be necessary in order to fully establish this point.

Further, the results herein described serve to strengthen the analogy existing between chlorine and bromine, and the difference in the temperatures necessary to enable the light to cause the combination of these elements with hydrogen is simply another rough measure of their relative affinity.

A more extended investigation of this subject will be undertaken, and exact measurements made of the rate of this change, at different temperatures, in the light and in the dark. The influence of light on the combination of hydrogen and iodine, at high temperatures, will also be studied.—*American Chemical Journal*, xx., No. 2.

A DELICATE TEST FOR THE DETECTION OF A YELLOW AZO DYE USED FOR THE ARTIFICIAL COLOURING OF FATS, &c.*

By JOSEPH F. GRISLER.

THE food laws of several of the States prohibit the use of colouring-matter in oleomargarine, and, inasmuch as the presence of artificial colouring-matter in many cases constitutes the only infraction of these particular laws, simple tests for its detection are desirable. Those engaged in clarifying and decolourising oils and fats are no doubt familiar with the use of Fuller's earth as a precipitant of colouring-matter, the latter usually being precipitated without any pronounced colour reaction. Since text-books and the chemical literature, to my knowledge, do not mention the red to pink colour which Fuller's earth produces with some of the yellow azo dyes, I desire to call attention to the practicability of using this earth as a most convenient and delicate test for the detection of at least one, if not more, of the yellow azo dyes. The latter are now largely used as constituents of butter-colours, and replace annatto to a considerable extent.

While analysing a sample of a reddish-coloured butter intended for export, my attention was drawn to the large amount of colouring-matter present and the intense violet-red colour developed when I attempted to remove the colouring-matter from the clarified fat by means of Fuller's earth. The colour developed depended much upon the relative amount of Fuller's earth added, ranging from a deep violet-red to pink. All the colouring-matter giving this particular reaction was readily precipitated. The precipitate, after having been thoroughly washed with naphtha to remove the fat, presented, upon drying, a violet-red powder. Contact with alcohol immediately decolourised it, the colour reappearing upon the evaporation of the alcohol. Boiling alcohol readily extracted all the colouring-matter from the precipitate, producing a

yellow solution. As obtained from the latter, the colouring-matter was found to be quite insoluble in water, sparingly soluble in hot water, re-precipitating from the latter on cooling. It dissolved with a yellow colour in concentrated sulphuric acid which, upon dilution with water, developed a bright pink to red colour. Strong mineral acids also produced a violet to pink colour. The latter reaction is familiar through the use of methyl orange as an indicator in acidimetry. True methyl orange as supplied in the market for use as an indicator, however, does not give the Fuller's earth reaction, nor is it as soluble in fats as this particular colouring-matter. These reactions with others would indicate one of the yellow azo dyes. The reactions throughout corresponded closely with a commercial yellow azo dye soluble in fats and used as a constituent of butter-colours.

Applying the Fuller's earth test to various samples of butter and oleomargarine, I found a large number of the samples to give the Fuller's earth reaction even where the samples were but lightly coloured.

In view of the fact that in certain localities oleomargarine was readily sold on the strength of the statement that only "two ounces of colouring-matter had been used per ton," and that the amount was "too small to admit of detection," it became a matter of interest to determine the limit of the Fuller's earth test and the probable amount of colouring-matter present in commercial samples of average colour. With this end in view mixtures were made with varying amounts of the yellow azo dye and pure white lard, and the test applied to the same. The mixtures were as follows:—

	Colouring-matter per kilo of lard.	Ratio.	Equivalent per ton. Ounces.
A. ..	100 m.grms.	1 : 10,000	3·2
B. ..	50 "	1 : 20,000	1·6
C. ..	10 "	1 : 100,000	0·32
D. ..	1 m.grm.	1 : 1,000,000	0·032

The latter ratio indicates fourteen grains per ton. Treated with Fuller's earth—

- A developed a deep violet-red colour.
- B developed a deep violet-red colour.
- C developed a deep pink colour, and
- D developed a pink tint.

In the latter case a half grm. of the sample spread upon a white porcelain slab developed a strong unmistakable pink upon the addition of Fuller's earth. When D was dissolved in naphtha and the Fuller's earth added to the solution, the pink appeared as a distinct ring or zone at the edge of the deposited layer of the reagent. The reaction is therefore a very delicate one.

Of the mixture A the average of several experiments showed that so minute a quantity as the 1/200th of a m.grm. still gave a pink reaction perceptible to the naked eye. Under the microscope the 1/100th part of this was still perceptible. Since the amount of colouring-matter actually present under the latter conditions was only 1/200,000,000 of a m.grm. (equivalent to 1/13,000,000,000 of a grain) the applicability of the test as a definite colour reaction under ordinary conditions is apparent. The use of as little as two ounces of the colouring-matter per ton produces a highly-coloured oleomargarine, while fourteen grains (the 10/625 part of two ounces) per ton would be barely perceptible as a faint yellow tint, but readily detected as a pink tint developed by the test.

Commercially, the yellow azo dye is generally used in conjunction with an orange variety. The latter does not give the Fuller's earth test.

For practical purposes the test is readily applied by spreading some of the clarified fat to be tested upon a white porcelain surface, and stirring into the fat a pinch of Fuller's earth and observing the change in colour. A pink to violet-red colour will appear within a few moments

* Read at the Washington Meeting of the American Chemical Society.

if any considerable proportion of this colouring-matter is present. If the experiment is performed in a glass tube it is readily preserved for court exhibits where such are desirable. The test may therefore be used as a valuable adjunct in testing for this colouring-matter in fats as well as in differentiating between certain of the azo dyes.

DISCUSSION.

C. A. CRAMPTON.—I wish to call attention to the very valuable and *appropos* nature of this test. It seems to be a very good one, and is especially valuable because this form of colouring-matter seems to have driven out of use the old-fashioned butter colour which was made of annatto. The manufacturers say the dyes "hold up" better than annatto, by which is meant, I suppose, that they will stand time and exposure to light much better. In the paper to be presented to-morrow, I hope to show some samples of this butter-colour dye, which will illustrate the extent to which it is used in a certain class of butters.

J. F. GEISLER.—It is generally claimed that these azo dyes are not detrimental to health. They are certainly used in very minute quantities.

E. A. DE SCHWEINITZ.—A number of physiological experiments have recently been made to determine the effect of these dyes, and, as a result, it is stated that they are not poisonous even when used in considerable quantities.—*Journ. Amer. Chem. Soc.*, vol. xx., No. 2, 1898.

LIQUID AIR BY THE GALLON.

At the Meeting of the Alumni Association of the Stevens Institute of Technology a very interesting exhibition of "liquid air" was made by Mr. W. H. Dickerson, M.E., representing Chas. E. Tripler, Esq., who is the inventor of the apparatus and process by which this remarkable product is readily prepared in large quantities, and who has developed the same at his own expense.

The liquid air (about 2 gallons in quantity) was brought from the works at 121, W. 89th St., New York City, very much as a can of milk might have been transported; that is, the liquid was in a zinc can placed in a wooden bucket, such as is used in transporting ice-cream, the space between the can and bucket being filled with ordinary hair-felt, such as is used in covering steam-pipes.

From this tin can the liquid air was dipped out as required with a tin ladle, exactly as milk would be served from an ordinary milk-can.

The condition of the liquid air is in fact this:—When produced it is of necessity intensely cold, because its critical temperature being about -189° F. no amount of pressure will liquefy it unless it is at least as cold as this.

When the pressure is relieved by drawing off the liquid into an open vessel, a portion at once vapourises, but in so doing cools the rest to a still lower temperature; and when so cold as this the tendency to vapourise is very moderate, so that, if the liquid air is placed in a vessel with non-conducting walls (like the tin can packed with felt in a wooden pail), it will only evaporate slowly, so that a quantity may be kept in this way for six or eight hours with only a trifling loss.

Among the interesting experiments performed on this occasion were the following:—

Liquid air was dipped out of the can and poured upon some mercury, which was promptly frozen, so that it could be shaped by hammering, exactly as a block of lead could be.

A piece of soft rubber tubing, dipped for a moment in the liquid air, became as brittle as glass, and was shattered into splinter-like fragments when struck by a hammer.

Wrought iron, cooled in like manner by liquid air, became so fragile that it was snapped with ease between

the fingers, but copper and aluminum lost none of their flexibility.

An egg or piece of meat frozen by this intense cold crumbled in the hand like a piece of cracker or dry bread.

A mass of cotton or of hair-felt soaked with liquid air and lit with a match burned violently, like meal gun-powder, or like the pyrotechnic mixtures used in many "fire-works."

Poured between the poles of a powerful magnet, the liquid air clung to the poles as would iron filings, and presently formed a bridge across the space between the poles. This was due to the magnetic properties of the oxygen of which air, in part, consists.

A tea-kettle filled with liquid air boiled, giving off clouds of apparent steam without being heated, and was even made to boil more violently by the application of ice. This last will be readily understood if we remember that, as compared with liquid air, ice is a very hot substance. The appearance of steam which was seen coming from the spout of the tea-kettle or elsewhere from the liquid air, was due to the condensation of the moisture in the general atmosphere by the intense cold of the "gaseous air" into which the "liquid air" was changing.

The most impressive feature of this exhibition was the large amount of liquid air present and the ease with which it was handled.

HENRY MORTON, Ph.D.

[It is interesting to note that experiments with liquid air can now be exhibited in the United States on so large a scale as our correspondent has above described. The experiments, however, are quite familiar to those of our readers who have attended Professor Dewar's lectures at the Royal Institution during the past ten years].

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, February 17th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

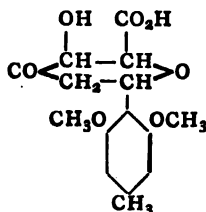
(Concluded from p. 103).

*19, "A Chemical Examination of the Constituents of Indian and American *Podophyllum*." By WYNDHAM R. DUNSTAN, F.R.S., and T. A. HENRY.

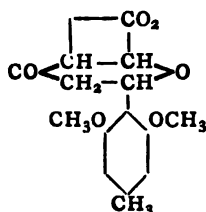
The authors find that the constituents of Indian *podophyllum* (*Podophyllum emodi*) and of American *podophyllum* (*Podophyllum peltatum*) are identical. The chief constituent is the *podophyllotoxin* of Podwysoski and Kürsten which the authors have fully examined. It is a neutral crystalline substance (m. p. 117°), to which the authors assign the formula $C_{15}H_{14}O_6$. It is strongly laevorotatory, and acts as a powerful purgative and intestinal irritant. When heated with alkalis, it is converted by hydration into the salt of an unstable gelatinous acid, *podophyllic acid*, $C_{15}H_{16}O_7$, of which a crystalline sodium salt was obtained, and also silver and copper salts, which were analysed. This acid very readily loses water, and furnishes the crystalline *picropodophyllin* of Podwysoski and Kürsten, which is isomeric with *podophyllotoxin*. It passes again into *podophyllic acid* when warmed with aqueous alkalis. It melts at 227° , and is optically inactive. *Podophyllotoxin* and *picropodophyllin* furnish identical decomposition products; when oxidised with nitric acid, *oxalic acid* is the principal product; when fused with alkalis *orcinol* and *acetic acid* are produced. Both substances contain two methoxyl groups and no hydroxyl. It is concluded that *picropodophyllin* is the

lactone of podophyllic acid, which is probably the hydroxy-carboxylic acid of *dimethoxymethyl-phenylhydro-γ-pyrrhons*.

The following formulæ are assigned to these compounds:—



Podophyllic acid.



Picropodophyllin.

The nature of the isomerism of podophyllotoxin and picropodophyllin remains to be determined. The latter substance is therapeutically inert.

The yellow colouring matter of podophyllum, called by Podwysoski podophylloquercetin is proved by the authors to be identical with *quercetin*, the valuable yellow colouring matter of quercitron bark.

An uncrystallisable resin, *podophylloresin*, was also isolated and found to exert a purgative action.

Estimations have been made of the amount of "podophyllin" (a mixture of resins with podophyllotoxin which is used in medicine) contained in the two plants. Indian podophyllum contains from 9 to 12 per cent, and American from 4 to 5 per cent. The two resins have been proved to be equally valuable therapeutic agents. The amount of crystalline podophyllotoxin in the Indian plant varies from 2 to 5 per cent, whilst representative samples of the American rhizome were found to contain rather less than 1 per cent.

Indian podophyllum is likely to be valuable both as a drug and as a dye-stuff.

DISCUSSION.

Mr. E. J. MILLARD asked if Professor Dunstan had noticed and could account for the smaller solubility of the resin from *P. emodi*. This was specially marked in the presence of a small quantity of an alkali when it assumed a gelatinous consistency. Since attention had been drawn to this resin by Professor Dunstan, manufacturers had prepared it in considerable quantities, and it was known commercially as the less soluble variety.

Professor DUNSTAN said, in reply, that any difference that may be observed in the solubility and other properties of podophyllin resin prepared from Indian podophyllum and that prepared from American podophyllum was probably accounted for by the different proportions in which the constituents were mixed in the resins prepared from the two sources and not by any difference in the constituents themselves.

*20. "The Volatile Constituents of the Wood of *Goupia tomentosa*." By WYNDHAM R. DUNSTAN, F.R.S., and T. A. HENRY.

Goupia tomentosa is a large tree growing in British Guiana, where it is known as "kabucalli." The wood is hard, and is used in the colony for boat building. When freshly cut it emits a smell resembling that of valerian. By distilling the wood with water, a mixture of acids of the acetic series was obtained, from which the authors have isolated and identified *formic acid*, *isovaleric acid*, *normal caproic acid*, and *lauric acid*. A small quantity of *succinic acid* was also obtained.

*21. "Oxycannabin from Indian Hemp." By WYNDHAM R. DUNSTAN, F.R.S., and T. A. HENRY.

Oxycannabin is the name given by Bolas and Francis to a crystalline substance they obtained by acting on the pharmacopœial extract of Indian hemp with concentrated nitric acid (*Trans.*, 1869, xxii., 417; *CHEM. NEWS*, 1871, xxiv., 77). They obtained it in the form of yellow needles

melting at 176°, and represented its composition by the formula $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_7$. In the course of the examination of the constituents of Indian hemp, the authors have prepared the substance in this way, and have also obtained it by the action of fuming nitric acid on the "cannabinol" isolated by Messrs. Wood, Spivey, and Easterfield (*Trans.*, 1896, lxix., 554) from the "charas" or resin of Indian hemp.

When quite pure, oxycannabin crystallises in colourless needles melting at 182°; it is insoluble in water, but soluble in hot alcohol, crystallising out on cooling. It sublimes when gently heated, and may be easily purified in this way. On combustion, the substance gives numbers agreeing with those required by the formula $\text{C}_{10}\text{H}_{10}\text{NO}_4$.

It does not dissolve in aqueous alkalis unless warmed with them in a closed tube. By acidifying the resulting solution, an acid is precipitated which is at present under investigation. "Oxycannabin" would therefore appear to be a lactone.

On reduction with hydriodic acid, or with tin and hydrochloric acid, oxycannabin furnishes a volatile amine. The hydrochloride of this base, though itself unstable, gives a well-crystallised platinichloride.

When heated with zinc dust, oxycannabin gives off a large amount of an inflammable gas (probably methane), and a quantity of an aromatic hydrocarbon is produced, which forms a well-crystallised compound with picric acid.

By the action of nitric acid on cannabinol, *normal butyric acid* was also produced, together with *oxalic acid*.

As the substance is thus proved to be a nitro-compound, the name "oxycannabin" is inappropriate, but it would be premature to propose a new name until more is known about the constitution of the compound.

In the course of purifying cannabinol through the acetyl derivative mentioned by Messrs. Wood, Spivey, and Easterfield, the acetyl derivative was obtained in colourless crystals melting at 75°, which corresponded in composition to the formula $\text{C}_{15}\text{H}_{23}\text{AcO}$.

DISCUSSION.

The PRESIDENT remarked that a similar investigation of the products of "oxycannabin" had been carried on for some time in the University Laboratory at Cambridge by Messrs. Spivey and Easterfield, and that they had isolated substantially the same products.

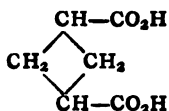
22. "On the Condensation of Formaldehyd with Ethylic Malonate, and on Cis- and Trans tetramethylenedicarboxylic Acids (1:3)." By E. W. HAWORTH and W. H. PERKIN, jun.

In a paper published some time since, (*Ber.*, 1886, xix., 1053), it was shown that when formaldehyd condenses with ethylic malonate in the presence of acetic anhydride, the principal product of the reaction is ethylic propane-tetracarboxylate, $(\text{CO}_2\text{Et})_2\text{CH}\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{Et})_2$, but that at the same time a substance boiling at 208—212° is produced in small quantities which, from the results of the analysis, was thought at the time to be ethylic methylenemalonate, $\text{CH}_2\cdot\text{C}(\text{CO}_2\text{Et})_2$.

In further investigating this reaction with large quantities of material, these results have been confirmed. The condensation product contains the following substances.

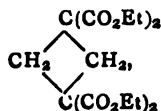
1. *Ethylic methylenemalonate*, $\text{CH}_2\cdot\text{C}(\text{CO}_2\text{Et})_2$, distils at 208° as a colourless oil which, on standing, rapidly polymerises into a hard wax-like substance, which is sometimes transparent and sometimes opaque. The polymeride, which is probably identical with the substance obtained by Zelinsky (*Ber.*, 1889, xxii., 3294), by the action of methylene iodide on ethylic malonate, on distillation yields small quantities of ethylic methylenemalonate, and considerable quantities of high-boiling oils which are at present under investigation.

When hydrolysed with alcoholic potash, the polymeride yields cis-tetramethylenedicarboxylic acid (1:3)—



2. An oil boiling at 234–236° (80 m.m.). Ethylic propanetetracarboxylate, $(\text{CO}_2\text{Et})_2\text{CH}\cdot\text{CH}_2\cdot\text{CH}(\text{CO}_2\text{Et})_2$.

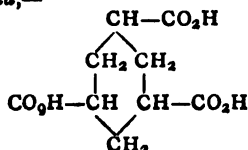
3. An oil boiling at 250—300° (40 m.m.), which evidently contains *ethylic tetramethylenetetra-carboxylate*,—



since, on hydrolysis, an acid is obtained which, after elimination of carbon dioxide and etherification, yields *ethyl cis-tetramethylenedicarboxylate* (1:3) (b. p. 172° at 50 m.m.). This ethereal salt, on hydrolysis, yields pure *cis-tetramethylenedicarboxylic acid* melting at 131°.

4. When the fraction 250–300° (40 m.m.) is treated as above, it yields besides ethylic cis-tetramethylenedicarboxylate, an ethereal salt boiling constantly at 202° (30 m.m.), and from this, on hydrolysis, a crystalline acid is obtained melting at 114°.

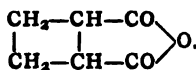
This acid, which on analysis gives numbers agreeing well with the formula $C_9H_{12}O_6 \cdot H_2O$, is probably *hexahydrotrimesic acid*.—



and in order to determine whether this view is correct, the acids formed by reducing trimesic acid are being investigated. The authors have already succeeded in preparing a crystalline tetrahydrotrimesic acid, $C_6H_4(CO_2H)_2$, which melts at about 185°, and is characterized by being very sparingly soluble in ether, but excessively soluble in water.

During the course of this research, Markownikoff's work on the action of dry sodium ethylate on ethylic α -chloropropionate has been carefully repeated (*Journ. Russ. Chem. Soc.*, 1890, xxii., 285; *Ann.*, 1881, ccviii., 333). It is found that both the cis- and trans- modifications of tetramethylenedicarboxylic acid (1 : 3) are formed in this way, and not only the trans-modification as Markownikoff states. The trans-acid (m. p. 171°), on treatment with acetyl chloride, yields the anhydride of the cis-acid (m. p. 50—51°), and this, on treatment with water, is readily converted into the cis-acid (m. p. 131°).

It is remarkable that Markownikoff, who first observed this behaviour, should have assumed that the trans-tetramethylenedicarboxylic acid, during this series of reactions, undergoes molecular change, and that the anhydride melting at 50–51° is the anhydride of tetramethylenedicarboxylic acid (1:2), *i.e.*,—

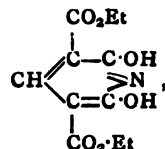


It will be shown in the detailed description of the experiments that Markownikoff's view, which has caused a good deal of confusion in the chemistry of the tetramethylenecarboxylic acids, is certainly incorrect.

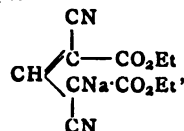
The authors are also engaged in investigating the action of methylene iodide on the disodium derivative of ethylic propanetetracarboxylate, and hope to be able soon to communicate the results of their experiments to the Society.

23. "Formation of Ethylic Dihydroxydinicotinate from Ethylic Cyanacetate." By S. RUHEMANN, Ph.D., M.A., and K. C. BROWNING, B.A.

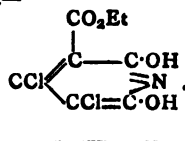
The authors give an account of their examination of the mother-liquor from the preparation of ethylic sodio-dicarboxylglutamate. They succeeded in isolating from it ethylicdihydroxydinicotinate.—



and attribute the formation of this compound to the presence of ethylic cyanacetate in the ethylic malonate. In order to verify this explanation, they studied the action of chloroform on ethylic cyanacetate in the presence of sodium alcoholate. They thus obtained ethylic sodium-dicyanoglutaconate,—



and prepared from it other metallic compounds and the ethereal salt itself (compare Errera, *Gas. Chim. Ital.*, 1897, xxvii, 333). On boiling the ethereal salt with dilute hydrochloric acid, ethylic dihydroxydinicotinate (m. p. 202°) is formed. Chlorine transforms the pyridine-derivative into ethylic dichlorodihydroxynicotinate, to which, from its behaviour towards phenylhydrazin, they assign the formula—



ROYAL INSTITUTION.

General Monthly Meeting, February 7th, 1898.

**Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer
and Vice-President, in the Chair.**

The following were elected Members:— Miss Cecilia Ash, Mrs. Henry C. A. Baynes, Miss M. E. Bevington, The Hon. Edith M. Boscawen, Miss Alice M. Burton, The Rev. J. J. Coxhead, A. C. Cronin, R. C. Forster, Dr. William Garnett, H. Godsall, A. H. Goschen, Major-General Coleridge Grove, C.B., A. Humbert, J. J. Kincaird, Capt. Wm. N. Lister, L. M. Lowenstein, G. W. Marriott, Mrs. E. R. Merton, T. Middlemore, B.S. Ogle, P. E. Singer, G. P. Taylor, J. Thornton, and Sir Arthur Spencer Wells, Bart.

The special thanks of the Members were returned to Mr. Hugh Leonard for a donation of £50 to the Fund for the Promotion of Experimental Research at Low Temperatures.

Melting-points of Silver and Gold.—D. Berthelot.—By means of a platinum-iridium thermo-electric cell the author has found the melting-point of silver to be 962° on an average of six experiments, and that of gold 1064° in the same manner. The first measurements of the melting-point of silver made with any degree of accuracy were by Pouillet in 1836, who made it 1000° . Becquerel in 1862 made it 960° , and that of gold 1092° . Other observers have been very near these figures, the greatest variations being only 102° .—*Comptes Rendus*, cxxvi., No. 6.

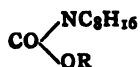
CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 6, February 7, 1898.

Transparency of Bismuth in a Magnetic Field.—H. Buisson.—According to the electro-magnetic theory of light the transparency of bodies is closely related to their electric resistance. It seemed interesting to try, on the same metal, if varying the conductivity also modified the transparency in the manner which might be expected. The variation of the resistance of bismuth in a magnetic field was utilised by the author. Very thin layers of bismuth were obtained by the electrolytic deposition of the metal on silvered glass, but, in spite of the action of a magnetic field of 15,000 C.G.S. units, which increased the electrical resistance by at least 60 per cent, no change in the transparency could be detected.

The Aromatic Urethanes of Conicine.—P. Caze-neuve and M. Moreau.—The energetic action of piperidine, which immediately gives urethanes by its reaction on phenolic carbonates, made it seem probable that conicine or propyl-piperidine would behave in a similar manner. Its action is, however, much less energetic; it does not give off any heat when mixed with the aromatic carbonic ethers, but requires to be boiled for the reaction to take place. By this means, and using two molecules of conicine to one molecule of carbonic ether, the authors have succeeded in preparing a number of liquid, uncrystallisable urethanes, with a viscosity increasing with the molecular weight; they are stable, and distil almost without alteration at the normal pressure; they correspond to the formula—



R being an aromatic radical. They are all saponifiable with potassic alcohol, and are decomposed by SO_4H_2 , giving off CO_2 .

A New Coloured Reaction of Phenylhydrazine.—Louis Simon.—The solution of phenylhydrazine is warmed for some instants with a few drops of aqueous trimethylamine; a few drops of a solution of nitroprussiate of soda are then added, followed by strong potash. Immediately on the addition of the nitroprussiate a clear blue colour appears, which is decidedly deepened by the addition of the potash. A small quantity of acetic acid modifies the colouration. The reaction enables one to detect 1/50,000th of phenylhydrazine. It is very distinct with 1/100,000th, but disappears after about a quarter of an hour.

Polymorphism of Fluorite.—F. Wallerant.—The author has found three distinct varieties of crystals of fluorite from different sources. The first is cubic-holohedric, the second is ternary, and the third binary.

No. 7, February 14, 1898.

A Crystallised Hydride of Dicumphene.—A. Etard and G. Meker.—Good results have been obtained by the action of sodium on the chlorhydrate of turpentine under the following conditions:—To 100 parts of dry chlorhydrate of turpentine, heated *only* to the temperature necessary to melt it, we add 15 parts of sodium, all at once, and shake frequently so as to granulate the melted sodium, and thus place it in good contact with the liquid chlorhydrate. It is of great importance to carry out this operation slowly, so as to prevent any rise of temperature favourable to excessive polymerisations. This control of temperature gives the best chance of a good return. The action of the sodium being finished, instead

of distilling in the presence of salt and a little sodium we add benzene to dissolve the carbides. The benzenic solution is washed with water, and then distilled. After the benzene is driven off we separate about 45 per cent of the liquid boiling between 150° and 160° , and 30 per cent boiling between 320° and 330° , by fractional distillation. The light fraction, made liquid by a little benzene, is shaken up with a little sulphuric acid; this acid should contain a lot of anhydride. In this manner it is easier to destroy the foreign carbides, and the hydride of cumphene remains alone. Wash with a filter-pump on glass wool, with fuming sulphuric acid, then with ordinary sulphuric acid until complete decolouration, and finally with water. On distillation we obtain from 15 to 20 per cent of a magnificently crystallised substance.

Action of Cyanamide on Bromanile in the Presence of Potash.—H. Imbert.—If to a boiling aqueous solution of cyanamide holding powdered bromanile in suspension we add a few small pieces of potash, the liquid takes a beautiful green colour in transmitted light, but is yellow to reflected light. The addition of an acid, such as hydrochloric, changes the colour first to blue and then to violet.

Researches on Organic Phosphorus.—L. Jolly.—The author tried to find whether integral metalloidal phosphorus existed in the organic molecules of animal tissue, or in albumenoid vegetable matters, but his results show that such is not the case.

Rapid Detection and Estimation of Manganese in Plants and Vegetable Earths, by a Colorimetric Method.—P. Pichard.—(See page 108).

Formation of Anhydride by the Calcination of Gypsum at a High Temperature.—A. Lacroix.—In a recent communication the author described the formation of anhydrous sulphate of lime, by heating gypsum to a relatively low temperature. He has since endeavoured to transform these new sulphates into anhydrite. At a dull red-heat there is no change; but, commencing at a cherry-red heat, the layers of the dehydrated gypsum become less coherent, and when pressed by the finger forms a crystalline powder; the same happens when immersed in water. On close examination it is seen that the material has undergone a complete change, and is completely transformed into anhydrite. If the temperature is carried still higher, the anhydrite melts and re-crystallises in large plates, even when rapidly cooled. Barytine (BaSO_4) and celestite (SrSO_4) both re-crystallise in the same manner with the greatest ease.

Justus Liebig's Annalen der Chemie,
Vol. ccc., No. 1.

Bromsuccinic Acids.—W. Lossen.—Kekulé found that by boiling solutions of dibromsuccinic acid salts in water they decompose, metallic bromides being formed, and another product, the nature of which depends on the base. When sodium, barium, or silver salts were used, the decomposition takes place according to the following equations:—

- (1). $\text{C}_4\text{H}_4\text{Br}_2\text{O}_4 + \text{H}_2\text{O} = \text{C}_4\text{H}_5\text{BrO}_5 + \text{HBr}$.
Monobrommalic acid.
- (2). $\text{C}_4\text{H}_4\text{Br}_2\text{O}_4 = \text{C}_4\text{H}_5\text{BrO}_4 + \text{HBr}$.
Monobromomaleic acid.
- (3). $\text{C}_4\text{H}_4\text{Br}_2\text{O}_4 + 2\text{H}_2\text{O} = \text{C}_4\text{H}_6\text{O}_6 + 2\text{HBr}$.
Tartaric acid.

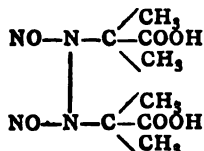
These three reactions were investigated by Lossen, with Reich, Mendthal, Riebenschalm, and Schwarzenberger. They found that dibromsuccinic acid decomposes according to the equation—



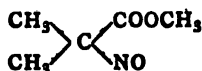
The bases potassium, barium, and calcium were used, but in no case was a reaction like that of Kekulé found to take place. The silver salt was not found to decompose in the same way as the others. The various reactions appeared to depend on the quantity of water with which the salt was boiled, but not at all or very slightly on the nature of the base. Kekulé's theory that the calcium salt is formed by boiling dibromsuccinic salts with water, which crystallises with 3 molecules of water, was proved to be incorrect. The acid formed was always a mixture of mesotartaric and racemic acids.

Hydrocinchonine.—O. Hesse. — Hydrocinchonine, which is always present in small quantity in commercial cinchonine prepared from the cinchona bark, occurs in much larger quantity in the bark of *Remijia Purdieana*. The cinchonine can be obtained pure by precipitating the mixture of cinchonine and hydrocinchonine with platinum chloride, as the cinchonine salt is crystalline, while the hydrocinchonine salt is flocculent. The alkaloid is obtained from the salt by precipitation with NH_3 , and is then crystallised from alcohol. The following salts can be prepared:—(1). $(\text{C}_{19}\text{H}_{24}\text{N}_2\text{O})_2 \cdot \text{PtCl}_6\text{H}_2$ as a yellow flocculent precipitate, from neutral platinum chloride; the precipitate changes to orange-coloured needles. (2). $\text{C}_{19}\text{H}_{24}\text{N}_2\text{O} \cdot \text{PtCl}_6\text{H}_2$ from acid platinum chloride. (3). Four neutral sulphates crystallising with $12\text{H}_2\text{O}$, $9\text{H}_2\text{O}$, $6\text{H}_2\text{O}$, $2\text{H}_2\text{O}$ respectively.

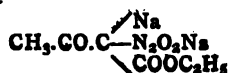
Isonitramin and Nitroso-iso-butyric Acid.—M. Gomberg.—When nitrous acid acts on hydrazo-*i*-butyric acid a very explosive nitrosamine is formed, which, as it is decomposed by alkalis into isonitramin-*i*-butyric acid and oxyisobutyric acid, very probably possesses the constitution—



The decomposition products were isolated and estimated at lead salt and zinc salt respectively. The isonitraminic acid is beautifully crystalline and melts at 94° to 95° . It is best obtained by decomposing the lead salt suspended in ether by means of H_2S . The action of nitrous acid on hydrazo-*i*-butyric ester gives a product which often explodes even when mixed with ice, while the nitrate solution is being added; it must therefore be violently agitated. When this is done it decomposes without danger into N and the ester of nitroso-*i*-butyric acid—

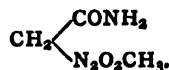


Synthesis of Nitrogen Compounds by means of Nitric Oxide.—Wilhelm Traube.—This paper contains a summary of the author's work on the action of NO on a large number of compounds. He finds that all the substances which react with nitrous acid to form isonitroso-compounds also absorb NO. The substances thus produced are all strong acids, and as their sodium salts are insoluble in alcohol, these are readily obtained as the first products of the synthetical method. This consists in dissolving the original substances in absolute alcohol, adding sodium ethylate, and passing NO into the solution, when either 4 or 4 molecules are directly added on to 1 molecule of the ketone. For example, when NO is led into a solution of aceto-acetic ether in alcohol which contains 2 molecules of sodium methylate to 1 molecule of ether, the crystalline di-sodium salt of isonitramin-aceto-acetic ether is precipitated—



The acids generally form salts with the heavy metals which are slightly soluble in water, and the acids when warmed with the mineral acids decompose, yielding β -substituted hydroxylamines.

Etherification of Isonitramine Fatty Acids.—H. Sielaff.—This paper is a special part of the previous one. The isonitraminic acids are transformed into silver salt and treated with alkyl iodide. Concentrated alcoholic NH_3 transforms these esters into crystalline amides such as—



The amide of methylisonitramin acetic acid, m.p. 142° ; of methylisonitramin propionic acid, m.p. 150° ; of methylisonitramin butyric acid, m.p. 126° . By saponification of the esters are obtained methyl-*o*-ethylisonitramin acetic acids, as syrups very soluble in water, and benzylisonitramin acetic acid, m.p. 135° .

MISCELLANEOUS.

The Estimation of Phosphorus in Steel.—R. W. Mahon (*J. Am. Chem. Soc.*, xix., 792–795).—The author describes a rapid method, successfully used with steels low in carbon and silicon and free from arsenic, which, he claims, will afford accurate results in about eight minutes. The procedure includes the partial neutralisation of the nitric acid and precipitation of the phosphomolybdate at a temperature near the boiling-point, and after shaking for fifteen seconds. The precipitate and filter are placed in a measured excess of caustic alkali; and the excess is determined by standard acid, with phenolphthalein as an indicator.

Reservoirs of Water in the Soil.—We take the following interesting paragraph from the *Agricultural Journal of the Cape of Good Hope* of January 20th, 1898:—"Nearly all the moisture used by plants is brought to them by the silent force of capillarity. If the soil is in proper physical condition, moisture flies upwards to the roots of vegetation from the great reservoirs in the subsoil as easily and as certainly as it runs downward by gravitation. The more the subject is studied the deeper is found the real underlying principles of successful husbandry. There should be an ample reservoir in the soil for the storage of moisture to tide over plants at critical periods. This moisture should not be in the form of free water, or that which is capable of being pushed along by its own weight, but water that is held in the soil by capillarity. Well-prepared soils are capable of holding about 30 per cent of their own weight of moisture by capillarity, and yet not contain any free or flowing water. Water may rise fully 3 feet by capillary force alone. Three feet of soil weigh 5400 tons per acre; 30 per cent of this is 1620 tons or 3,622,800 lbs., or 400,000 gallons. This is Nature's great reservoir from which plants draw moisture. If this reservoir is but 6 inches deep—the more common depth—it will hold but one-sixth as much, and hence plants grown over this small reservoir would likely droop in dry weather. Ample reservoirs, secured by means of under-drainage, culture, and tap-rooted plants, bid defiance to any reasonable drought. Having provided a supply of water, the next effort is to make as much of it as possible pass through the plant and allow as little as possible to evaporate from the surface. This is done by keeping the soil so porous and loose for 2 or 3 inches at and near the surface that water cannot pass upwards except through the plants. Beneath our feet, then, are found ever acting, kindly forces, and unnumbered forms of vegetation, all waiting to be guided and directed into channels by the skill of the husbandman."

MEETINGS FOR THE WEEK.

- MONDAY, 14th.**—Society of Arts, 8. (Cantor Lectures). "The Thermo-chemistry of the Bessemer Process," by Prof. W. N. Hartley, F.R.S.
- Society of Chemical Industry, 8. "Electrical Industries at the Foyers Waterfalls," by R. W. Wallace, Q.C.
- TUESDAY, 15th.**—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
- Society of Arts, 4.30. "The West Indies and Sugar Bounties," by Neville Lubbock.
- WEDNESDAY, 16th.**—Society of Arts, 8. "The Recent History of Paper-making," by Clayton Beadle.
- THURSDAY, 17th.**—Chemical, 8. "The Reduction of Bromic Acid, and the Law of Mass Action," by Winifred Judson, B.Sc., and J. Wallace Walker, M.A., Ph.D. "The Action of Ferric Chloride on the Etheral Salts of Ketone Acids," by R. S. Morell, M.A., Ph.D., and J. M. Crofts, B.A., Ph.D. "Note on the Volatility of Sulphur," by T. C. Porter. "Action of Ammonia and Substituted Ammonias on Acetylurethane," by George Young, Ph.D., and Ernest Clark.
- Royal Institution, 3. "Recent Researches in Magnetism and Diamagnetism," by Prof. J. A. Fleming, M.A., D.Sc., F.R.S.
- FRIDAY, 18th.**—Royal Institution, 9. "The Bringing of Water to Birmingham from the Welsh Mountains," by James Mansergh, V.P. Inst. C.E., F.G.S.
- SATURDAY, 19th.**—Royal Institution, 3. "English Letter Writers," by Professor Walter Raleigh, M.A.

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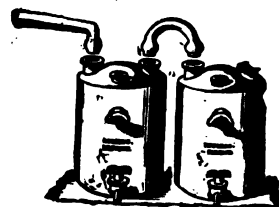
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THE CHEMICAL NEWS.

VOL. LXXVII., No. 1999.

PREPARATION OF LEAD-FREE AMMONIUM SALTS OF TARTARIC AND CITRIC ACIDS.

By L. DE KONINGH, F.C.S.

WARINGTON has stated that when estimating lead in tartaric or citric acid of commerce, the comparison liquid should contain the same amount of either of these acids, as otherwise the results will be quite untrustworthy. The difficulty is, however, to procure these acids free from lead or copper.

When using Warington's process (*Journ. Soc. Chem. Ind.*, 1893, 97-101) there is no absolute need for the pure acids, as in reality their ammonium salts are used. The writer, not having succeeded in procuring metal-free samples, has made some experiments to obtain lead-free ammonium salts, with the following results:—

By Crystallisation as Acid Tartrates.—Fifty grms. of tartaric acid were dissolved in 300 c.c. of water, and two drops of a 10 per cent solution of lead acetate were added. A little more than one half of the solution was now neutralised with ammonia, and the remainder was then added; this caused an immediate deposit of acid ammonium tartrate, which after a while became more dense and crystalline. The mother-liquor, on adding ammonia and hydrogen sulphide, gave, as might have been expected, an intense reaction for lead; but it soon appeared that the lead was far from being all in solution; then washing failed to completely remove it, and even after a litre of water had been used the washings still gave a strong lead reaction. This is very strange; for although lead tartrate is a very insoluble salt, it is said to be readily soluble in neutral ammonium tartrate, and the mother-liquor contained a large quantity of this salt. A portion of the crystalline mass was now squeezed dry between filter-paper, then dissolved in dilute ammonia and again tested, when it was found to be strongly contaminated with lead. The remainder was now dissolved in boiling water, and it separated on cooling in heavy well-formed crystals; the mother-liquor gave an intense lead reaction, and the crystals were therefore once more re-crystallised. It was now expected they would be pure, but they were still so contaminated with lead as to be perfectly unfit for use in Warington's process.

Application of Egeling's Process.—Egeling (*Ned. Tydschr. Pharm.*, 1896) has published a very handy process for the detection of traces of lead in waters. The sample is treated with hydrogen sulphide, and if any colouration should make its appearance the liquid is agitated with a little kaolin, which carries down the merest traces of lead or copper sulphide.

The writer has found this to be the best plan of freeing a solution of ammonium tartrate or citrate from lead and copper. To the solution, which should be strongly alkaline, a little strong hydrogen sulphide is cautiously added until the colour does not get any darker; a little kaolin is then added, and the whole well stirred until it has combined with the sulphides. The liquid is now filtered, and should at once be used for the comparison test. If the filtrate is yellowish, this may be remedied by agitating with purified animal charcoal. The filtrate should then once more be tested with hydrogen sulphide and, if necessary, be again shaken with kaolin.

The writer doubts whether hydrogen sulphide really removes all the lead; but, at all events, Warington's instructions that the comparison liquid should consist of an ammoniacal solution of either citric or tartaric acid,

unaffected by ammonium sulphide, are properly carried out.

Efforts were made to obtain pure ammonium-hydrogen tartrate from a solution of tartaric acid strongly contaminated with lead and copper and treated by the kaolin method. Acetic acid was added, and the precipitate, after slight washing by decantation, was re-crystallised from boiling water. The result was a complete failure; and when tested it gave an intense lead reaction. This, however, may have been derived from the glazure of the porcelain dish. Solutions of ammonium citrate freed from metals by means of the kaolin process and boiled down in a beaker for some time, also had taken up a large quantity of lead. No doubt it will act, in the long run, even in the cold, so that if prepared in large quantity and kept in stock it should be again tested before use.

Warington has noticed that the colour of lead sulphide shows up much stronger in a solution of citric acid than in plain water. If, therefore, pure ammonium citrate in a solid state was at disposal, might this not be successfully used in testing water for lead in cases where the mere adding of hydrogen sulphide fails to produce a colouration?

325, Kennington Road, S.E., March 11, 1898.

THE RECENT SANDSTORM OFF THE WEST COAST OF AFRICA.

By LEONARD DOBBIN, Ph.D.

THE daily papers of February 21st, in a telegram from Madrid, reported the occurrence at the Canary Islands, a few days before, of a sand-storm from the Sahara. This storm appears to have been experienced over a wide area. On the 15th February, when in lat. 22° 5' N., long. 17° 25' W., the R.M.S. *Roslin Castle* passed through the storm, and large quantities of fine sand fell upon the deck and adhered to the sails and rigging. By the kindness of Walter Berry, Esq., Danish Consul-General, a passenger on this steamer, who joined it at Madeira a few days later and succeeded in obtaining a specimen of the sand, a sample came into the hands of Professor Crum Brown, who handed it to me for examination.

The sample consisted of an extremely fine reddish-brown powder, which, on microscopic examination, was found to contain a large proportion of nearly white siliceous-looking particles. Treated with dilute hydrochloric acid, there was a considerable evolution of carbonic anhydride, and, on boiling for some time with concentrated hydrochloric acid, a bright yellow solution and a nearly white insoluble residue were obtained. The residue consisted essentially of silica, and a quantitative experiment showed that it amounted to 53·8 per cent of the whole.

The hydrochloric acid solution was examined for metals, and was found to contain iron, aluminium (in very small quantity), calcium, magnesium, potassium, and sodium.

The salt radicals found (in addition to silica and the salt radical of the carbonates) were those of the chlorides, sulphates (small quantity), and orthophosphates (traces only).

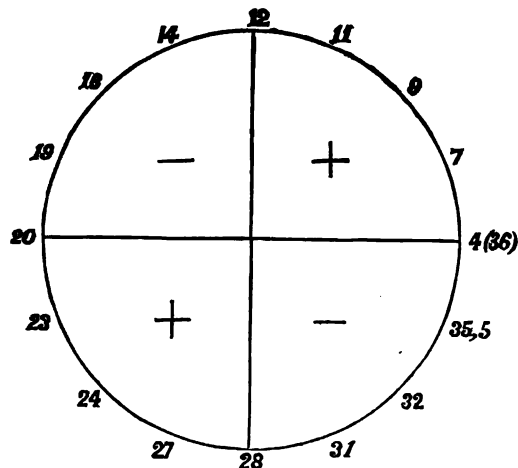
No traces of manganese or of borates could be detected. Dried at 100° the powder lost 2·7 per cent of moisture. An aqueous extract from the original powder possessed a marked alkaline reaction, indicating the presence of alkali metal carbonate.

It is unnecessary to comment upon the unpleasant nature of a shower of this fine alkaline dust.

Mr. Berry has been good enough to add the following particulars:—

The R.M.S. *Roslin Castle*, Capt. de la Cour Travers, entered the dust cloud 5—10 miles off Cape Blanco, and

atomic weights:—We see at once that the elements of the first and third quadrant are electro-positive, but those of the second and fourth electro-negative. We have the strongest electrical character in the elements which lie nearest the horizontal diameter, i. e., attach themselves to an inactive element, 4 (36) or 20, consequently in this case to lithium and chlorine, sodium and fluorine, whilst carbon and silicon—with an undecided electrical character—attach themselves to the perpendicular diameter.



This behaviour reminds us, without doubt, of the function $\cotang. x$, and if we call the atomic weight a , the electro-chemical character, ϵ , of all the elements of this group may be expressed by—

$$\epsilon = \cotang. \frac{a-4}{16} \pi.$$

As a matter of course this formula cannot express the absolute value of the electrical character of the elements, but only the general course of its dependence on the atomic weight.

With the increase of the atomic weight the valency of the elements is altered; it rises in the first quadrant from null to a maximum,—e. g., 4,—passes back to null in the second, reaches its maximum again in the third quadrant, and returns finally in the fourth to null. The rise and fall of the valency with the growing atomic weight of the elements is consequently expressed by the + and — marked in the quadrants.

These considerations also give us information on the mathematical expression for the dependency of the valency on the atomic weight. Such a course would be given in its simplest form by $m (\sin x)^2$ if m is the maximum of the valency hence designated by 4 in the above group of elements. Hence we may assume that the formula—

$$v = 4f \left(\sin \frac{a-4}{16} \pi \right)^2,$$

if f denotes a function of the square of the same which would express the rise and fall of the valency for all the values of a situate between 36 and 4.

For the second main group of the elements with atomic weights between 36 and 132 (potassium to iodine) it is easily intelligible that a similar relation would appear between the atomic weight on the one hand, and the electrical character and the valence of the elements on the other, only that the constants would obtain a different value. The angle must be expressed by—

$$\frac{a-36}{48} \pi.$$

Possibly there would be substituted an elliptical arrange-

ment of the elements for the circular. In the same manner must be conceived the relation of the elements of the third main group with atomic weights from 132 to 152, of which hitherto only the first half is partially known. Concerning the first small group with atomic weights from 0 to 4, it must be assumed that only includes hydrogen, which would be in harmony with the small atomic weight (4) of first inactive element.

By the assumption of the existence of the inactive elements here in question with atomic weights 4, 20, 36, 84, 132, 212, and 292, with an indifferent electro-chemical character and a valence = 0, and by the arrangement of the elements which I have indicated, the periodicity in the property of the elements appears as a continuous function, and at the same time we perceive the cause of the arrangement in groups each of two series, since each group corresponds to the four quadrants of the circular arrangement. We may therefore assume that by these supplements the periodic system has received a form which will essentially facilitate a future mathematical treatment of the entire problem. — *Zeitschrift für Anorganische Chemie*, vol. ix., p. 283.

ON THE SPECTROGRAPHIC ANALYSIS OF SOME COMMERCIAL SAMPLES OF METALS, OF CHEMICAL PREPARATIONS, AND MINERALS FROM THE STASSFURTH POTASH BEDS.

By WALTER NOEL HARTLEY, F.R.S., and
HUGH RAMAGE, F.I.C., A.R.C.Sci.L., Royal College of Science,
Dublin.

As already published (*Proc. Roy. Soc.*, 1896, lxx., p. 393, and *Trans.*, 1897, p. 281), we have found gallium to be a common constituent of many iron ores, and have separated it from blast-furnace metal smelted at Middlesbrough. Lately, we have been led to examine the steel obtained from the same metal by the basic Bessemer process, and rolled into rails. Considering that alumina in a very high degree of purity is prepared from Irish bauxite and reduced to aluminium in the electric furnace, we have carefully examined samples kindly supplied us by the British Aluminium Company, Limited, of (1) pure alumina from the Company's works at Larne, Co. Antrim (the process of preparation has been described by Mr. James Sutherland, *Engineering*, lxii., p. 291); (2) the residual ferric oxide, or "red mud"; and (3) commercially pure aluminium, containing about 99.75 per cent of the metal.

Alumina and Aluminium.—In 1, the pure alumina, we found sodium, potassium, copper, gallium, iron, lead, and traces of indium. In 2, the "red mud," sodium, potassium, calcium, copper, silver, gallium, iron, nickel, chromium, and lead. No. 3, the metallic aluminium, contained sodium, potassium, calcium, copper, silver, gallium, iron, manganese, lead, and traces of indium.

It will thus be seen that a trace of iron, and some of the silver, copper, and gallium, pass from the bauxite, which is obtained from mines at Glenravel, Co. Antrim, into the very carefully prepared alumina manufactured at Larne, and finally makes its appearance in the aluminium reduced at Foyers, Inverness-shire.

The following remarks may be aptly made in connection with the results obtained:—First, the aluminium is not stated to be present, because it gives a spectrum in the oxyhydrogen flame only under certain conditions which are not always under control, and as the aluminium was known to be the chief constituent of the majority of the substances, it was not necessary always to state it as such. Silica and titanous acid are not recognised by this method of examination,

Secondly, all the results were obtained in exactly the same way from a weighed quantity of each substance.

Thirdly, the alkali metals, calcium, iron, and copper, are not substances accidentally present in the flame, since we have made a variety of experiments at different times, and have no difficulty whatever in obtaining spectra free from iron and copper. We have no greater difficulty in obtaining spectra free from potassium and calcium lines, and with those of sodium so reduced as to be nothing more than a mere suggestion of the element being in the flame.

It will thus be understood that every element mentioned is actually a constituent of the metal or compound examined. It is of importance that this should be noted.

Railway Metal.—We made an examination of drillings from steel rails from the North Eastern Steel Co.'s works at Middlesbrough. They contained sodium, potassium, calcium, copper, silver (a trace), gallium, manganese, and lead (a trace).

It is interesting to note that steel rails may contain small quantities of metal not hitherto suspected of being alloyed in commercial iron of such purity, as, for instance, copper, silver, gallium, and lead. Nickel and chromium, although contained in the crude, or "mixer metal," from the blast furnaces, are present in more minute proportions in the steel made by the basic Bessemer process.

The systematic examination of railway metal by such an analytical method as this would prove of much interest, and might become of actual practical importance.

Salts of Aluminium.—In endeavouring to trace the passage of the rarer metals from aluminous ores into various commercial salts of alumina, we made experiments on various laboratory samples of alum and crude aluminoferric cake.

These we will describe as follows:—

- | | |
|------------------------------|------------------------|
| 1. Ammonium alum, crystals. | 5. Granulated alum. |
| 2. Potassium alum, crystals. | 6. Turkey-red alum. |
| 3. Ground alum. | 7. Aluminoferric cake. |
| 4. Ordinary lump alum. | |

No. 1, the ammonia alum, contained sodium, potassium, a trace of calcium, copper, gallium, thallium, and a trace of iron. No. 2, the potassium alum, contained, in addition to the metals known to be present, rubidium, thallium, a trace of iron, and nickel.

In each of the samples numbered 3, 4, 5, and 6, the following four metals were detected:—Sodium, rubidium, calcium, and thallium.

The rubidium lines were strong in the Turkey-red alum.

No. 7, the aluminoferric, contained sodium, calcium, copper, gallium, thallium, iron, nickel, a trace of manganese, chromium, and lead.

We considered that it was desirable that larger quantities of some of these salts should be operated upon, and the gallium separated by the method described by Lecoq de Boisbaudran, which consists in precipitating it as ferrocyanide by the addition of potassium ferrocyanide to a very strongly acidified solution of the salt made by adding three times its volume of concentrated hydrochloric acid to the aqueous solution. The purity of the ferrocyanide was first proved before being used for this test.

Fifty grms. of each of the samples Nos. 1, 2, 5, and 6 were tested.

The ferrocyanide precipitates were collected on filters of ashless paper and burnt.

The precipitate from No. 1 yielded silver, nickel, and a trace of manganese, in addition to the metals previously observed in this salt, but without the thallium, which was thus seen not to be carried down.

The other samples, which are potassium alums, all yielded precipitates containing sodium, rubidium, calcium, copper, gallium, thallium, and cesium.

Ten grms. of the aluminoferric, treated in like manner, yielded, in addition to the above-mentioned metals, nickel, a trace of manganese and lead.

It will be noticed that rubidium, cesium, gallium,

thallium, copper, nickel, and silver, are all thrown down by potassium ferrocyanide when it is added to a strongly acidified solution. Whilst the gallium comes from the aluminous ore, bauxite, the thallium comes from the sulphuric acid. We have detected it in the pyrites used, and with it a trace of indium.

Rubidium and cesium, it was conjectured, had been introduced with potash salt, and it was considered desirable to examine not only the sources of potash, but also other minerals obtained from the Stassfurth potash beds. The results gave an emphatic negative to this supposition.

The Stassfurth Minerals.

The following were examined:—

- Kainite, $\text{MgSO}_4 \cdot \text{KCl} + 3\text{H}_2\text{O}$.
- Tachydrile, $\text{CaCl}_2 \cdot 2\text{MgCl}_2 + 12\text{H}_2\text{O}$.
- Sylvine, KCl .
- Kieserite, $\text{MgSO}_4 + \text{H}_2\text{O}$.
- Stassfurthite, $\text{MgCl}_2 \cdot 6\text{MgB}_2\text{O}_4 \cdot \text{B}_2\text{O}_3$.
- Epsomite, $\text{MgSO}_4 + 7\text{H}_2\text{O}$.
- Rock salt.

Every specimen contained both sodium and potassium, but neither rubidium nor cesium. All but Stassfurthite contain calcium; all but sylvine and rock salt contain magnesium also. Iron is a constituent of all. Strontium and a trace of manganese are found in tachydrile; strontium and a trace of nickel in epsomite; boric oxide bands were very strong in the Stassfurthite spectrum, which also showed a trace of copper to be present.

We have no doubt whatever, now, that the rubidium and cesium come from the bauxite, whether Irish or French, and from shale, in which we have also found these elements associated with potassium and lithium. This fact is remarkable.

The shale used in the manufacture of the alums is the richest mineral in gallium, sodium, potassium, and rubidium, and poorest in nickel and silver. Specimens of Irish bauxite and shale have been found to be equally rich in copper, and French bauxite richest in lithium and silver.

To trace the cesium satisfactorily it would be necessary to work on larger quantities of material, and separate—at least partially—the other alkali metals, because the continuous spectrum given by these when present in large proportion masks the weaker lines of other elements.—*Transactions of the Chemical Society*, 1897.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING FEBRUARY 28TH, 1898.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, *Metropolis Water Act*, 1871.

London, March 10th, 1898.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 168 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from Feb. 1st to Feb. 28th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several

samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 168 samples examined by us, all were found to be clear, bright, and well filtered.

We have again to record a great deficiency in the rainfall; although rain has fallen at Oxford on sixteen days out of the month, the total amount is only 1.19 inches; as the average for the last thirty years is 1.76 inches, we have a deficiency of 0.57 inch on the month, bringing the present deficiency for the year to 1.91 inches.

Our bacteriological examinations of 232 samples have given the results recorded in the following table; we have also examined 31 other samples, from special wells, stand-pipes, &c., making a total of 263 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	490
New River, filtered (mean of 23 samples) ..	22
Thames, unfiltered (mean of 24 samples) ..	2119
Thames water, from the clear water wells of five Thames-derived supplies (mean of 114 samples)	24
Ditto ditto highest	258
Ditto ditto lowest	0
River Lea, unfiltered (mean of 24 samples) ..	1017
River Lea, from the East London Company's clear water well (mean of 23 samples) ..	23

The two Rivers supplying London with water are in remarkably good condition for the time of year. This is no doubt due to the abnormally low rainfall. The excellent condition of the filtering and storage works of the Companies is shown by the high quality of the water revealed by the enclosed analyses.

We are, Sir,

Your obedient Servants,

WILLIAM CROOKES.
JAMES DEWAR.

ON A COMBINATION OF PHOSPHORIC ANHYDRIDE WITH BENZENE.

By H. GIRAN.

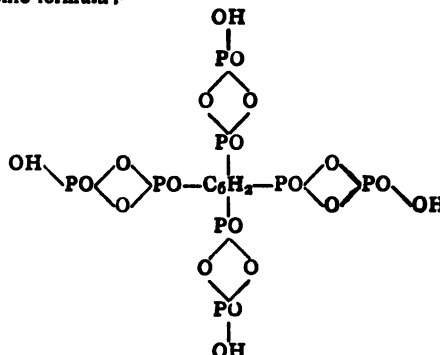
THE formation of sulphonic acids by the action of fuming sulphuric acid on various organic compounds, led me to think that phosphoric acid might also give rise to bodies analogous to these sulphonic acids. For the purpose of verifying this hypothesis in one particular case, I heated a mixture of two parts of phosphoric anhydride with three parts of benzene (or one molecule of P_2O_5 and 3 molecules of C_6H_6) for four or five hours in a sealed tube at from 120° to 120° . A solid brick-red, but very unstable, substance was produced, which dissociated rapidly in free air, but which could be preserved in an atmosphere of benzene. In the presence of water it decomposes into benzene and phosphoric acid, but it is soluble in alcohol. This solution slowly attacks carbonate of baryta, and changes after the lapse of a few hours into a gelatinous mass. The same result is achieved if we replace the carbonate of baryta by an alcoholic solution of ethylate of baryta until the solution is neutral. This gelatinous mass is thrown on a filter, washed with absolute alcohol, and dried on a porous plate in the presence of caustic potash.

We thus obtain a white salt soluble in water, which, after desiccation in the hot-air oven at a temperature of from 120° to 120° , gives on analysis a composition corresponding to the formula $C_6H_2P_8O_{20}Ba_2$. The results of the analysis are—

	Calculated.	Found.				
		I.	II.	III.	IV.	V.
C	7.86	8.02	7.95	—	—	—
H	0.21	0.36	0.28	—	—	—
P	27.07	—	—	27.67	26.86	—
Ba	29.92	—	—	—	—	30.57

The acid of this salt should be $C_7H_2P_8O_{20}H_4$; it should result from the union of one molecule of benzene with four molecules of phosphoric anhydride ($C_6H_6 + 4P_2O_5$).

Its constitution may be explained by the following graphic formula:—



This should thus be *benzenetetradimetaphosphoric acid*.

I propose to study the properties of this acid and its salts in detail, and to try and isolate various combinations—which theory enables me to predict—formed by the union of the anhydride or of the phosphoric acids with benzene or with other organic compounds of the same order.—*Comptes Rendus*, ccxvii., No. 8.

SODIUM PEROXIDE IN QUANTITATIVE ANALYSIS.*

By C. GLASER.

W. HEMPEL (*Ztschr. Anorg. Chem.*, 1892, iii., 193-194) and J. Clark (*J. Chem. Soc.*, 1893, 1079) first proposed to use sodium peroxide in quantitative analysis. Hempel employed it for the oxidation of chromium, manganese, tungsten, and tin, and subsequent determination by known means; he also mentions that sulphur is completely oxidised to trioxide. He further recommends the reagent for the decomposition of zinc-blende and galena.

J. Clark recommends it for the estimation of sulphur, arsenic, and chromium, also for the separation of manganese from zinc, nickel, and cobalt. He states (*Ibid.*, lxiii., 1079) that the action of sodium peroxide on coke and coal is too violent for analytical purposes. T. Spüller and S. Kalman (*Chem. Ztg.*, xvii., 18) use it on ferrochrome, chrome steel, chrome iron, and also sulphurets. Poleck (*Chem. Ztg.*, xviii., 103) experimented on organic substances, as did J. Tafel (*Ber. d. Chem. Ges.*, xxvii., 816).

In 1894 M. Hoehnel and C. Glaser (*Arch. Pharm.*, ccxxii., 222; *Chem. Ztg.*, xviii., 1148) made revised recommendations for the estimation of sulphur in pyrites. O. Kassner (*Arch. Pharm.*, ccxxii., 226) states that iron is precipitated as hydroxide, but not changed into ferric acid,—that, on the contrary, solutions of ferric acid are reduced by sodium peroxide. He gives detailed statements concerning separation of chromium and manganese; of the decomposition of freshly precipitated sulphides of antimony, tin, and arsenic. In 1895, L. Archbutt (*Analyst*, xx.) reports an analysis of sodium

* A review of various propositions made since 1892.

peroxide, showing the presence of nearly one-half of 1 per cent of iron and alumina in the commercial article. Alb. Edinger (*Ztschr. Anal. Chem.*, xxxiv., 362) gives his experience in the determination of sulphur and chlorine in inorganic and organic combinations. M. C. Schuyler (*Chem. Ztg.*, xx., 1896) proposed to determine mercury in mercuric salts by reduction to metal with sodium peroxide. In 1897 S. W. Parr (*Y. Am. Chem. Soc.*, xix., 341) reports on the adaptability of sodium peroxide as a third group reagent in qualitative analysis, practically a review of the above-named publications, omitting quantitative features.

The *Chemiker Zeitung* (*Chem. Ztg.*, xxi., No. 6) contains a second note of the author, suggesting some improvements in the determination of sulphur in pyrites, and later the same author (*Ibid.*, xxi., No. 69) separated iron from aluminum in phosphates and other minerals by means of sodium peroxide.

It may be in place here to state that the C. P. sodium peroxide of the trade has, as far as I have had occasion to test it, proved quite free from oxide of iron and alumina. It appears therefore probable that the article analysed by Archbutt was a crude commercial product.

I cannot confirm O. Kassner's statement that iron is not oxidised to ferric acid by sodium peroxide. If the reagent is added to a solution of an iron salt, without the precaution of keeping the temperature of the latter sufficiently low, his statement holds good. But if this last condition is complied with by keeping the solution comparatively cool, a sufficient excess of the reagent will produce a perfect solution, as I have stated in the above last-named paper.

I desire especially to recur to J. Clark's statement that the action of sodium peroxide on coke and coal is too violent to permit of use in analysis. I have used this reagent in a considerable number of coke, coal, and asphalt analyses for sulphur with perfect success, and I now present a short description of the method as I carry it out.

The material to be analysed is placed in a sufficiently large silver or nickel dish, and covered with about four times its weight of sodium carbonate. Upon this a piece of sodium hydroxide is laid, about one-half the weight of the carbonate used.

The dish is now moved carefully and slowly over a small flame until the gas generation subsides and a half-sized mass is obtained. Upon this mass dry sodium peroxide is now dusted from a porcelain or platinum spoon in small quantities at a time, always waiting until the reaction is over. This is continued until all carbon is burned away, when the mass, if necessary, is heated to perfect fusion. Usually this is obtained by the heat generated by the combustion in the dish, which, however, takes place so quietly that no loss by explosion or spurting will occur. After this operation the determination of sulphur is carried to an end in the well-known way.

I believe that this method will prove quite a relief to those chemists who have to make many sulphur determinations in coke and coal, and who know the uncertain working of a fusion with potassium nitrate. At least it has been my experience that it is difficult to so regulate the temperature of the fusion as to avoid explosion with loss. Even the use of double crucibles has not always proved successful. The time required for the operation with sodium peroxide is quite short; with coke about five minutes, with coal probably ten, while with asphalt—owing to the peculiar nature of the material—it will take a little longer.

In analysis of chrome ores, sodium peroxide does not appear to fulfil the hopes of those chemists who first proposed it. The oxidation is reported to be never perfect in one operation, which makes the method more complicated and troublesome than a fusion with potassium nitrate and borax.

This was to be expected, since even chromic salts in

solution require more than one treatment with sodium peroxide before complete oxidation to chromic acid is effected.

With this exception sodium peroxide has proved a very useful and convenient reagent, applicable in a great number and variety of cases. It ought to be, and no doubt will become, one of the standard reagents of a modern laboratory.—*Journal of the American Chemical Society*, xx., No. 2, 1898.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 3rd, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

MR. CECIL J. BROOKES was formally admitted a Fellow of the Society.

Certificates were read for the first time in favour of Messrs. Joseph Brierley, B.Sc., Ashton Road, Failsworth, Manchester; Arthur William Cowburn, Fernroyd, Bowdon, Cheshire; Charles Benjamin Dudley, Ph.D., Attoona, Penn., U.S.A.; Arthur Leonard Harry Garaid, c/o C. Lawes and Co., Barking Creek; Lawrence Hislop, Gas Works, Uddington; F. Hurter, Ph.D., Holly Lodge, Cressington Park, Liverpool; Samuel Morton Jessop, 12, Hanson Terrace, Wakefield; George Henry Masson, M.D., M.S., 22, Lauriston Place, Edinburgh; Walter Charles Cross Peters, 14, Trinity Square, S.E.; Francis Pitt Smith, B.Ph., 77, Woodland Avenue, New Rochelle, N.Y., U.S.A.; Thomas de Smith, B.A., Eastbourne College, Eastbourne; William Thomas Newton Spivey, M.A., 5, Trumpington Street, Cambridge; John Alexander Williamson, 81, Cheverton Road, Upper Holloway, N.; Thomas Barlow Wood, M.A., Caius College, Cambridge.

Of the following papers those marked * were read:—

*24. "*Preparation of Anhydrous Hydrogen Cyanide and Carbon Monoxide.*" By JOHN WADE, B.Sc., and LAURENCE C. PANTING, M.B.

On allowing a cold mixture of equal volumes of sulphuric acid and water to drop on to 98 per cent "lump" potassium cyanide, hydrogen cyanide, accompanied by traces of water only, is evolved in almost theoretical amount, and with the aid of suitable condensing apparatus is readily collected in quantity. With concentrated sulphuric acid, on the other hand, but still at the ordinary temperature, nearly pure carbon monoxide is evolved, also in quantity approaching the theoretical; provided certain precautions are taken, it is free from dioxide, and is accompanied only by small quantities of hydrogen cyanide.

In discussing the mechanism of the formation of the monoxide, experiments are described showing that part of the water required for the hydrolysis is derived either from the sulphuric acid itself or from the potassium hydrogen sulphate formed in the course of the action, and that consequently the sulphuric acid acts at the same instant both as a hydrolyst and as a dehydrating agent.

*25. "*Production of some Nitro- and Amido-oxytutidines.*" By J. N. COLLIE, Ph.D., F.R.S., and THOMAS TICKLE.

In a former paper (*Trans.*, 1897, lxxi., 838) one of the authors drew attention to the fact that various nitro- and amido-derivatives of pyridine could be obtained by the ordinary process of nitration and reduction of certain oxypyridine compounds. These substances correspond in the pyridine series to nitro- and amido-phenols in the benzene series, and as some of the reactions (especially those with various oxidising agents) of the amido-oxy-

pyridines resemble those of alkaloids, the investigation has been continued.

Pseudolutidostyryl (α -dimethyl- α' -oxypyridine) gave on nitration a nitropseudolutidostyryl, $C_5H_7NMe_2(OH)NO_2$. It crystallises in light yellow needles that melt with much decomposition at about 250° . The hydroxyl- and nitro-groups are in the ortho-position to one another, but the substance is not volatile with steam. With alkalis, it forms brilliant yellow compounds.

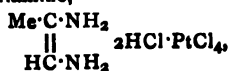
When the nitro-compound is reduced with tin and hydrochloric acid, an amidopseudolutidostyryl is produced. The hydrochloride of this base, $C_5H_7NMe_2(OH)NH_2HCl$, crystallises in needles which decompose without melting at $235-240^\circ$. By the action of sodium bicarbonate, the free base can be obtained in the form of a bulky mass of fine needles, m. p. 205° corr.). The base is very soluble in hot water, but much less so in cold. It oxidises readily, and an aqueous solution on boiling turns brown. This solution reduces silver nitrate at once. With ferric chloride it gives first a red and then a bright green colour, and when dissolved in strong sulphuric acid, if a drop of fuming nitric acid be added, a brilliant purple colour is momentarily produced. It forms a platinichloride and an acetyl derivative, m. p. 255° corr.).

By nitrating ethylic pseudolutidostyrylcarboxylate (m. p. $137-138^\circ$) a nitro-derivative was formed, $C_5N \cdot Me_2(OH)(CO_2Et)NO_2$. This substance, m. p. 215° corr.), crystallises in yellow needles. On hydrolysis, it yielded the corresponding acid, m. p. 260° corr.), which when pure is almost colourless, but forms bright yellow salts. The acid on heating loses carbon dioxide and the same nitro-pseudolutidostyryl (m. p. about 250°) was obtained.

Amido-pseudolutidostyrylcarboxylic acid was prepared from the corresponding nitro-acid by reduction. Its hydrochloride crystallises from water in needles with two molecules of water. The amido-acid melts at 275° corr.), and contains one molecule of water of crystallisation; above its melting-point it decomposes, yielding amidopseudolutidostyryl (m. p. 205° corr.). Its salts reduce silver nitrate solution, and with ferric chloride give a green colouration.

*26. "Production of some Nitro- and Amido-oxylutidines." (Part II.). By Miss L. HALL and J. NORMAN COLLIE, Ph.D., F.R.S.

Lutidone, $\alpha\alpha'$ -dimethyl- γ -oxypyridine, when warmed with nitric acid, does not yield a nitro-derivative (*Trans.*, 1897, lxxi., 838) but a nitrate of lutidone. If, however, a mixture of strong nitric and sulphuric acids are employed, the nitro-compound is formed, $C_7H_9NO + HNO_3 = C_7H_9NO \cdot NO_2 + H_2O$. This nitro-lutidone is a pale yellow crystalline compound, possessing a strong acid reaction, and dissolving in alkalis with an intense yellow colour; it is not volatile in steam. When reduced with tin and hydrochloric acid, it yields an amidolutidone, $C_7H_9NO \cdot NO_2 + 3H_2 = C_7H_9NONH_2 + 2H_2O$. This amidolutidone, unlike amidodioxypicoline and amidopseudolutidostyryl, does not give characteristic colours with oxidising agents, but acts as a strong reducing agent. It forms both a monohydrochloride and a dihydrochloride. The platinichloride is unstable; it undergoes reduction when dissolved in water and warmed, but if its hydrochloric acid solution is boiled, a very insoluble platinichloride separates, which seems to be the salt of propine diamine,—



the decomposition having been brought about by hydrolysis, $C_7H_{10}N_2O + 3H_2O = C_5H_7N_2 + 2C_2H_5O_2$. This breaking down of the pyridine ring is of considerable interest, and, in a substance like lutidone, is hardly to be expected, since the nitrogen atom is bound to two carbon atoms, neither of which are united to oxygen, and, moreover, lutidine is not the anhydride of an amido-acid.

DISCUSSION.

Mr. LING pointed out that certain anilides give, when treated with oxidising agents in acid solution, colour reactions similar to those obtained with alkaloids (Tafel, *Ber.*, 1892, xxv., 412; Schär, *Archiv. Pharm.*, 1894, ccxxiii., 249).

Prof. COLLIE, in reply to suggestions of Dr. Wynne, pointed out that it was impossible in the case of lutidone to obtain more than one mono-nitro-derivative. In nitrating pseudolutidostyryl, the position of the entering nitro-group had been determined with certainty by the fact that pseudolutidostyrylcarboxylic acid, after nitration and subsequent heating, yielded the same nitro-compound as had been obtained from pseudolutidostyryl itself.

*27. "On Benzene Hexabromide." By F. E. MATTHEWS, Ph.D.

In this paper, the author describes his failure to isolate the second modification of benzene-hexabromide, obtained, in minute quantity, by Orndorf and Howells (*Amer. Chem. Journ.*, 1896, xviii., 312-319) even from considerable quantities of the crude hexabromide; also the failure to prepare either the hexachloride or hexabromide of bromobenzene; in the former case owing to the displacement of chlorine by bromine, and in the latter to the substitution of bromine taking place more readily than direct addition.

The action of alcoholic soda upon benzene hexabromide is also described, the product of the action being a mixture of paradibromo- and tribromobenzene (1 : 2 : 4), possibly mixed with a small amount of one of the other dibromobenzenes. Benzene obtained from the hexabromide by nascent hydrogen in acid alcoholic solution yields both modifications of the hexachloride on treatment with chlorine.

DISCUSSION.

Professor TILDEN was glad that Dr. Matthews had resumed work on these interesting compounds. He had repeated some of Dr. Matthews's experiments, and had satisfied himself that the hexachloride was obtainable very readily and in very large proportion. It was somewhat remarkable that the corresponding bromide should be formed so much less readily.

Dr. MATTHEWS stated that he had always worked with material spontaneously precipitated from the mixture of benzene, bromine, and water, and not with the substance left after evaporating the mixture to dryness, as was the case with Orndorf and Howells.

*28. "Note on the Action of Bromine on Benzene." By J. NORMAN COLLIE, Ph.D., F.R.S., and COLIN C. FRYE.

The authors have repeated the work of Ador and Rilliet (*Ber.*, 1875, viii., 1286), who stated that when bromine was allowed to act on excess of benzene in sunlight, dibromo-addition products were produced, which, after treatment with zinc ethyl and subsequent oxidation with chromic acid, yielded metabromo- and metaphthalic-acids, together with benzoic acid, parabromobenzoic and terephthalic acid, but they were unable to obtain any ortho-compounds. The authors have succeeded in proving the presence of ortho-compounds, and at the same time have confirmed the observations of Ador and Rilliet, having isolated para-bromobenzoic acid and metaphthalic acid.

DISCUSSION.

Dr. MATTHEWS said that he had made several attempts to isolate di- and tetra-chlorine and bromine addition-compounds of benzene, both by the action of the halogens upon excess of benzene and also by decomposition of the hexabromide and hexachlorides of benzene, but always without finding a trace of any di- or tetra-halogen addition-derivative.

*29. "Notes on Manganic Salts." By C. E. RICE, B.A.

The author shows that the decomposition of manganic chloride in solution into manganous chloride and chlorine is reversible, the velocity of the reverse change being very small. He also describes the production and analysis of two double manganic chlorides, $2KCl \cdot MnCl_3 \cdot H_2O$ and

$2\text{NH}_4\text{Cl} \cdot \text{MnCl}_3 \cdot \text{H}_2\text{O}$, but obtains no evidence of the existence of any compound of the formula MnCl_4 .

DISCUSSION.

Professor CLOWES asked which of the manganese hydroxides was precipitated by the action of water upon the crystalline hydrated double chloride; it would be interesting to know whether a hydroxide corresponding to the formula MnCl_3 could exist.

Professor TILDEN remarked that the manganese tetrachloride seemed to be now finally disposed of, and in representing the production of chlorine by the usual process we should have to write a new equation.

Mr. RICE, in reply, said that he had not considered it necessary to investigate the point, since Pickering had shown that when the solution obtained by dissolving any of the higher oxides of manganese (Mn_2O_3 , Mn_3O_4 , MnO_2) in hydrochloric acid is diluted, the precipitate consists of a mixture of these hydrated oxides in varying proportions.

*30. "Some Chemical Properties of Concentrated Solutions of certain Salts. Part I. Potassium Carbonate." By W. COLEBROOK REYNOLDS.

When the salts of certain other metals are added to a concentrated solution of potassium carbonate, double salts are formed which are sometimes, as in the case of iron, copper, nickel, and cobalt, soluble in the solution, instead of the normal or basic carbonates which are formed when a dilute solution is employed.

These double salts and their solutions are decomposed by pure water. To obtain them, the chloride, nitrate, or preferably the acetate, is added to a concentrated solution of potassium carbonate (sp. gr. 1.55), and the liquid left to crystallise. The author has isolated the following double salts in well-defined crystalline form:— $\text{CuK}_2(\text{CO}_3)_2$, $\text{CuK}_2(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{MnK}_2(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{FeK}_2(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{CaK}_2(\text{CO}_3)_2$, $\text{Bi}_2\text{OK}_4(\text{CO}_3)_4 \cdot \text{H}_2\text{O}$, $\text{CoK}_2(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{NiK}_2(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{MgK}_2(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$, AgKCO_3 . The last four salts have been previously obtained by other chemists.

DISCUSSION.

Professor TILDEN said that Mr. Reynolds had been led to the study of these compounds in the course of enquiring into the production of the recently discovered percarbonates. In dilute solution, potassium carbonate appeared to be resolvable into the ions 2K and CO_3 , while in a saturated solution it behaved as though made up of K and KCO_3 . Mr. Reynolds had omitted to point out that in the formulae of all his new double carbonates, the heavy metal seemed to be associated with a proportion of KCO_3 corresponding to its valency. Perhaps this was only a coincidence, but it seemed to deserve notice.

Mr. A. G. BLOXAM remarked that if carbonic acid was regarded as a hydroxy-acid, the formation of a deep blue coloured solution by mixing a concentrated solution of potassium carbonate with a solution of copper sulphate became of interest as bringing carbonic acid into line with other hydroxy-acids, all, or nearly all, of which were known to give deep blue coloured solutions with copper sulphate and alkali sufficient to satisfy both the acidic and phenolic or alcoholic hydroxyl groups. Such solutions, however, were more stable than the potassium-copper carbonate solution of Mr. Reynolds, and this might be due to a comparatively unstable condition of potassium carbonate in solution; if a strong solution of this salt was partly decomposed on dilution, potassium hydroxide and bicarbonate being formed, a corresponding decomposition of the copper compound would be expected. He would be glad to learn from Mr. Reynolds whether addition of potassium hydroxide hindered the precipitation of copper carbonate on dilution.

Mr. W. P. BLOXAM asked whether the formation of these salts was attended with the evolution of carbon dioxide. Had the author taken into account the hydrolysis of potassium carbonate which took place in aqueous solution? According to a paper published by Senderens, the action of a non-metallic element (sulphur) upon solution

of potassium carbonate could be explained by regarding the salt as hydrolysed thus, $\text{H}_2\text{O} + \text{K}_2\text{CO}_3 = \text{KHCO}_3 + \text{KOH}$; and experiments made by the speaker showed that even a concentrated solution of the salt must be regarded as possessing this constitution. The presence of more oxygen in the double carbonate of bismuth than that present in the acid groups would be explained by the action of the hydroxide groups present in the carbonate solution. The same reaction would also explain the difficulty of obtaining manganous and ferrous double carbonates.

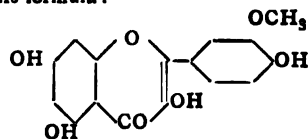
Mr. REYNOLDS, in reply, stated that it was possible to represent the formation of these compounds as taking place in accordance with such an equation as, for instance, $\text{MnCl}_2 + 2\text{K} \cdot \text{KCO}_3 = \text{Mn}(\text{KCO}_3)_2 + 2\text{KCl}$, and this view was strengthened by the fact that some of the salts, as, for instance, copper acetate, dissolved immediately without forming a precipitate which subsequently re-dissolved.

In the case of the double succinates, however, where exactly the same phenomena occurred, the composition of the copper compound was $\text{CuK}_4(\text{C}_4\text{H}_4\text{O}_4)_3 \cdot 2\text{H}_2\text{O}$, and this could not be represented by a similar equation. He had, therefore, not introduced such views into the present paper, as they did not appear to be generally applicable.

The author did not think that any of the phenomena supported the view that the solution contained potassium hydroxide and potassium bicarbonate, as no effervescence occurred, except in the case of normal bismuth salts, when basic salts were formed.

31. "The Colouring Matters of the Indian Dyestuff *Delphinium salii*." By A. G. PERKIN and J. A. PILGRIM.

"Aebarg" consists of the dried flowers and flowering stems of the *Delphinium salii*, found in great quantity in Afghanistan, and which is much used in India for the production of a yellow colour on alum mordanted fabrics. The flowering stems are nearly devoid of dyeing property. For the supply of material we are indebted to the authorities of the Imperial Institute. Three colouring matters exist in this plant in the form of glucosides. The sparingly soluble colouring matter, $\text{C}_{16}\text{H}_{12}\text{O}_7$, forms glistening yellow needles, soluble in alkalis with a yellow colour. Fused with alkali it yielded phloroglucinol and protocathechuic acid, and by means of hydriodic acid it yielded quercetin with the evolution of 1 mol. of methyl iodide. By methylation it was converted into quercetin-tetramethyl ether, and by acetylation into a tetracetyl derivative of the formula $\text{C}_{16}\text{H}_8\text{O}_7(\text{C}_2\text{H}_5\text{O})_4$, colourless needles, m. p. 195–196°. It was evidently *isorhamnetin*, a quercetinmonomethyl ether, recently isolated from the yellow wallflower, *Cheiranthus Cheiri* (Trans., 1896, lxix., 1650). As by oxidation in alkaline solution this yields vanillic acid, it has most probably the constitution represented by this formula:—



With alumina as mordant it dyes a purer yellow than quercetin. The chief constituent of the more soluble portion was recognised to be identical with *quercetin*, the colouring matter of quercitron bark. The residual colouring matter, present only in small quantity, was not obtained in a pure condition. It resembled quercetin in percentage composition, and in its decomposition products, but differed from it in not reacting with alcoholic potassium acetate, and the melting-point of its acetyl derivative. "Aebarg" resembles quercitron bark in dyeing property, but has only 35 per cent the tinctorial power of this dye-stuff. It contains, when freed from the flowering stalks, 3.47 per cent of colouring matter (not as glucoside).

32. "Some Metallic Salts of Natural Yellow Colouring Matters." By A. G. PERKIN and P. J. WOOD.

That certain colouring matters decompose alkaline carbonates with the formation of acid salts has been previously demonstrated. Thus of morin, Hlasiwetz and Pfaundler describe salts of this description, and of quercetin (*Fahresbericht*, 1864, 560). Further, from gentisin, Hlasiwetz and Habermann (*A.*, 1875, clxxv., 73) obtained analogous compounds. Among other instances is bixin, the colouring matter of annatto, which Etti describes (*Ber.*, 1878, xi., 864) as giving a sodium and a potassium salt, and rottlerin, from which one of us obtained similar compounds (*Trans.*, 1895, lxxvii., 230).

Owing probably to the formation of acid salts these alcoholic alkaline solutions are not readily neutralised completely by acetic acid, and as it appeared that some of the formulæ of the above compounds must be incorrect, this reaction was studied. It was found that in alcoholic solution, quercetin, morin, fisetin, and myricetin decompose potassium and sodium acetates with the formation of sparingly soluble salts. *Potassium quercetin*, $C_{15}H_9O_7K$ (found, $K = 11.46$; theory requires $K = 11.47$), forms minute orange-yellow prismatic needles, insoluble in cold water, and but slowly decomposed by boiling dilute acetic acid. *Sodium quercetin*, $C_{15}H_9O_7Na$, closely resembles the above salt. *Potassium morin*, $C_{15}H_9O_7K$ (found, $K = 11.97$; theory requires $K = 11.47$), separates as glistening orange-coloured needles, and is closely resembled by *sodium morin*, $C_{15}H_9O_7Na$ (found, $Na = 6.89$; theory, 7.09). Morin reacts with magnesium and ammonium acetates, though from quercetin salts have not been obtained in this way. *Ammonium morin* and *magnesium morin* separate in the form of long needles in a similar way (found, $Mg = 3.52$; theory requires $Mg = 3.83$ per cent).

Fisetin and myricetin apparently yield analogous derivatives, and on the other hand luteolin, apigenin, chrysin, and gentisin do not appear to react with alkaline acetates. With the exception of bixin and rottlerin, as yet but little examined, the above colouring matters are known to contain no carboxyl group, and it is thus of interest to study this reaction in which only one of the numerous hydroxyls present takes part. It appears probable that some analogy exists between these salts and the acid compounds of the colouring matters described by one of us (*Trans.*, 1896, 69, 1439).

33, "The Interaction of Magnesium and Solution of Copper Sulphate." By EDWARD DIVERS, M.D., F.R.S.

Neither Clowes and Caven nor Tilden, in their communications made to the Society in November last, seem to the writer to have recognised the significance of the results of the interaction of magnesium metal and a solution of copper sulphate. Remarkable as these results are, it is to be remembered that a closely analogous case had long been known, even when Commaille in 1866 observed them, namely, that of zinc immersed in a solution of an alum. Here, also, there is free evolution of hydrogen and precipitation of basic sulphate, and, when chromium alum replaces the aluminium salt, there is also reduction of some of the chromic sulphate to chromous sulphate. The only part of the change which finds no parallel in the action of zinc upon chrome alum is that of the deposition of a little copper, but this deposition is quite in accordance with the general behaviour of copper salts.

The formation of basic salt and hydrogen is a change independent of that of reduction, as is clearly shown by the same formation taking place, without that of reduction, when aluminium sulphate or common alum is concerned. Now, since the alum solution is dialysable into sulphuric acid and basic aluminium or chromium sulphate (besides potassium sulphate), and is also strongly acid in reaction, it will be admitted by all that the action in the case of an alum is really that of dilute sulphuric acid upon the zinc. The gradual precipitation of the previously soluble basic salt as the zinc dissolves in the solution is

just what happens when zinc sulphate is dissolved in a dialysed solution of aluminium, or chromium hydroxide, or basic sulphate. These two points being admitted, it will be very difficult to doubt that similar changes occur between copper sulphate and magnesium, or, in a less degree, zinc; for the solution is here also very acid in reaction and needs only a little boiling to make it deposit basic sulphate; besides, too, it will unquestionably show largely that hydrolysing action upon cane-sugar which Long (*Y. Amer. Chem. Soc.*, 1896, 18, 120) has recently shown so many metallic salts possess, and this property is evidence that, like aluminium sulphate, it is partly hydrolysed into sulphuric acid and soluble basic sulphate, which will be precipitated as magnesium goes into the solution. Caven and Clowes have pointed out the inadequacy of the action of the magnesium-copper couple to account for the large evolution of hydrogen. Their unsupported suggestion that the hydrogen is formed by the combined action of copper sulphate (as such) and magnesium upon water, has likewise little claim to consideration in presence of the fact that hydrolysed aluminium sulphate acts in the same way as copper sulphate.

Coming now to the production of cuprous oxide, a separate reaction, or set of reactions, there would be nothing in this unlike that of the reduction of ferric or chromic sulphate by zinc or magnesium, could cuprous salt only be found in solution. When, in place of the sulphate, cupric chloride is used, then, as everyone knows, there is just this looked-for reduction to cuprous salt. But the peculiarity of copper is that oxylic cuprous salts are apparently unable to exist. It is, however, not so improbable that in dilute solution and in presence of much cupric sulphate a little cuprous sulphate may exist for a very short time. According, indeed, to Rose, from roasted copper sulphide water extracts, along with cupric sulphate, a little cuprous sulphate, which then gradually decomposes into metal and cupric salt, just as a mercurous salt in water will change into metal and mercuric salt (*Annalen*, 1841, 39, 109). However that may be, and the observation admits of another interpretation, it does seem to the writer that the precipitation of cuprous oxide during the action of magnesium upon cuprous sulphate is a fact highly favourable to the view that cuprous sulphate is actually formed, part of it then quickly decomposing into cupric sulphate and metallic copper, and the rest of it being decomposed by the basic cupric salt into normal cupric sulphate and cuprous oxide. With certainty he can state that, when finely divided copper is acted upon by nitrogen peroxide, nitric oxide and a copper nitrate are formed, and no nitrite, and that this copper nitrate, when touched with water, decomposes into cupric nitrate and bright metallic copper, thus proving, apparently, that in absence of water cuprous nitrate can exist, and, therefore, by analogy, other oxylic cuprous salts.

There remains only to notice Tilden's suggestion that some of the hydrogen reduces cupric to cuprous oxide. Hydrogen is known not to have such an action, and therefore the usual assumption of there being a more active "nascent" hydrogen has to be made, but also without the least foundation in experience. In the present case, it may be pointed out, firstly, that in the reduction of chromic sulphate to chromous sulphate by zinc, hydrogen continues to be evolved, although very great excess of chromic salt is always in contact with the zinc; and, secondly, that in the reduction of ferric salts by zinc it has been established that this takes place much more rapidly when clean zinc dust is put into the ferric solution in the absence of excess of acid, than when, as is usual, an excess of acid is added to generate hydrogen. It may, therefore, with great probability be assumed that the reactions are $2CuSO_4 + Mg = Cu_2SO_4 + MgSO_4$ and $Fe_2(SO_4)_3 + Zn = 2FeSO_4 + ZnSO_4$, hydrogen having nothing to do in the matter.

PHYSICAL SOCIETY.

Ordinary Meeting, March 11th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

PROF. J. D. EVERETT gave a communication on "Dynamical Illustrations of certain Optical Phenomena."

The first part of the paper deals with the properties of a series of equal particles attached at equal intervals to an uniform stretched elastic weightless string. Their free simple-harmonic modes of wave-motion are first investigated. The highest frequency occurs when the wave-length is double that of the common distance a . As the wave-length increases from $2a$ to infinity, or diminishes from $2a$ to a , the frequency tends to zero. To every wave-length λ_1 between $2a$ and infinity, there corresponds a wave-length λ_2 between $2a$ and a , such that—

$$\frac{a}{\lambda_1} + \frac{a}{\lambda_2} = 1.$$

The frequency is the same for λ_2 as for λ_1 . Further examination shows that the difference of wave-length between these two solutions is only apparent, and that, so far as the movements of the particles are concerned, waves of length λ_1 travelling in one direction, are identical with waves of length λ_2 travelling in the opposite direction. The same is true if $a/\lambda_1 + a/\lambda_2$, instead of being unity, is equal to any integer. On the other hand, if the difference between a/λ_1 and a/λ_2 is an integer, the two sets of waves travel in the same direction. Any simple-harmonic wave-motion of the system of particles may thus be regarded as having any one of an infinite number of wave-lengths. When one particle of the system is constrained to a S.H. motion, of frequency not exceeding that which corresponds to $\lambda = 2a$, the whole system will ultimately vibrate in equal waves. When the frequency of the constrained particle exceeds that due to $\lambda = 2a$, the ultimate state will be S.H. motion with exact opposition of phase between successive particles. The simultaneous displacements of the particles at any instant, as we travel away from the constrained particle in either direction, form a diminishing geometrical progression, with signs alternately plus and minus. Expressions are investigated for the constraining force, and for the ratio of the energy of the system (consisting of an unlimited number of particles) to the energy of the constrained particle.

The second part of the paper deals with pendulums. (1.) Sympathetic pendulums, such as two equal pendulums suspended from the same support. (2.) Double pendulums, i.e., one simple pendulum suspended from another. In each case the investigation consists in seeking a mode of vibration in which the two bobs have either identical or opposite phases, so that their displacements are in a constant ratio, positive or negative. In every case there are two such modes, one with a positive and the other with a negative ratio. As regards the sympathetic pendulums:—When they are equal in mass and length the periods for the two modes are approximately equal, and the displacement of each pendulum follows the law of a "curve of beats"; the excursions are largest for one pendulum when they are smallest for the other. As regards the double pendulum:—When the lower mass is much less than the upper there exist, in like manner, motions following the law of beats, provided that, to start with, one bob is at rest in the zero position, and the other at rest in an extreme position. If the lengths of the two strings are decidedly unequal, one fundamental mode has approximately the period of the upper, and the other the period of the lower pendulum. In the former, the displacements of the two bobs are comparable; in the latter, the displacement of the upper is small compared with the lower. The bearing of these conclusions is pointed out, first, on Lord Kelvin's conclusions respecting a suspended clock; and, secondly, on Lord Rayleigh's assertion (frequently quoted in connection

with anomalous dispersion) respecting the influence on a heavy pendulum of a much lighter one suspended from it. To obtain the phenomenon of beats in perfection the upper string must be slightly longer than the lower, and the ratio of difference to sum of lengths must equal the ratio of lower mass to upper. The beats thus obtained explain the experiment described in the second edition of "Rayleigh on Sound," § 62. Sellmeier's application of the beats of double pendulums to explain fluorescence is briefly described. Stokes explains fluorescence by the analogy of the chain of equal particles discussed in the first part of the paper. Forced vibrations quicker than the critical frequency are produced by the action of the vibrating ether on the fluorescent body; and when the body is left to itself its subsequent motion is made up of S.H. components, all of which are below the critical frequency.

Prof. R. A. LEHFELDT then read a paper on "The Properties of Liquid Mixtures."

In a previous communication (*Phil. Mag.*, [5], vol. xl., p. 398) the author follows out the consequences of a certain thermo-dynamic relation between the composition of a liquid mixture and that of the vapour in equilibrium with it, and the saturation-pressure of the system. More stable compounds are now chosen, viz., benzene and toluene mixed with carbon tetrachloride, as types of normal organic compounds; and benzene and toluene mixed with ethyl alcohol as types of a so-called "associated liquid."

These experiments have been carried out in the Davy-Faraday Laboratory.

The measurements come under two distinct groups:—(1) Vapour-pressure; (2) composition of vapour. They were made separately, on material from the same source, prepared identically. To measure the vapour-pressure of the mixtures the "dynamic" method was adopted. An experiment consists in weighing out a mixture, taking its refractive-index by a Pulfrich refractometer, placing it in a boiling tube, and, after adjusting temperature and pressure, taking observations at different temperatures on a rising scale, and then on a falling scale. The refractive index of the residue is again measured; this is always used for checking the composition of the mixtures. For determining the composition of the vapour over liquid mixtures, the method used is to distil a little of the mixture and analyse the distillate. The apparatus is arranged so that the distillate can be drawn off by a tap as required.

The author criticises the results of Linebarger (*Journ. Amer. Chem. Soc.*, vol. xvii.), and also those of Margules (*Wein. Ber.*, vol. civ.). Linebarger states that the partial pressure of benzene and toluene in mixtures is simply proportional to the molecular percentage present. This conclusion, the author considers, is only roughly true; the partial pressure of the hydrocarbon vapour is not necessarily linear in mixture. Hence, the rule proposed by Linebarger for determining the molecular weight is incorrect.

The PRESIDENT proposed votes of thanks to the authors, and the meeting was adjourned until March 25.

CHEMICAL NOTICES FROM FOREIGN SOURCES

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

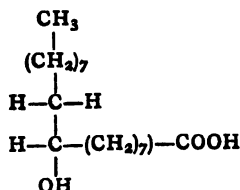
Journal für Praktische Chemie,
No. 1, 1898.

Chlorine and Bromine Derivatives of β -Naphthylamines.—Ad. Claus and O. Jäck.—This paper contains a

description of the preparation and properties of a large quantity of chlor- and brom-derivatives of β naphthylamines.

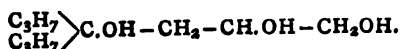
The Liquid Fatty Acids of the Fat of Dogfish.—Eugen Ljubarsky.—The fat of the Caspian dogfish was found to be a reddish liquid with a disagreeable fatty smell. If it was left in a vessel open to the air it became covered with a skin, darker in colour and smelling very bad. This fat when examined was found to consist of 17 per cent of saturated acid, in all probability palmitic acid. The remaining 83 per cent consisted of oleic acid, $C_{18}H_{34}O_2$, and physeloleic acid, $C_{16}H_{30}O_2$.

Action of Sulphuric Acid on Elaidic Acid.—Alex. Tscherbakoff and A. Saytzeff.—A theory is put forward that oleic and elaidic acids are stereo-isomers. In confirmation of this it is found that by treatment with sulphuric acid and potash both yield the same oxystearic acid, having the constitution—



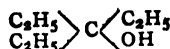
Theoretically oleic and elaidic acids should yield two different oxystearic acids, related as racemic and meso-tartaric acid, but it is suggested that in oxystearic acid one form only is stable.

The Trivalent Alcohol of Allyldipropylcarbinol.—Alexis Bogorodsky.—This substance was prepared by the action of a dilute solution of potassium permanganate on allyldipropylcarbinol, the permanganate solution being added in small quantities and kept cool. The glycerin was extracted with ether and dried in a vacuum desiccator. It was shown to have the constitution—



An attempt to prepare the acetic ether of the glycerin by heating the glycerin with acetic anhydride in sealed tubes was unsuccessful; apparently some quantity of diacetyl-ether derivative was formed.

Methyldiethylethylene.—Mich. Saytzeff, jun.—This substance was prepared by distilling a mixture of triethylcarbinol—

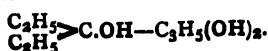


and oxalic acid. It boils at $97^\circ-98^\circ$ and has a specific gravity of 0.72544 at 15° . It has the constitution—



An attempt to oxidise it with potassium permanganate to the corresponding glycol gave only negative results, fatty acids of low molecular weight being the chief products.

Allylethylphenylcarbinol.—Al. Bogorodsky and J. Ljubarsky.—The method employed for the preparation of this substance was the action of allyliodide and zinc upon ethylphenylketone. The liquids were dropped from a dropping-funnel upon finely-powdered well-etched zinc. The oily product was separated from the water, dried over melted potash, and then distilled. The purest fraction obtained had a boiling-point of $238-242^\circ$. It was a mobile highly refractive liquid, insoluble in water and of lower specific gravity. An attempt was made to transform it into the corresponding glycerol by means of potassium permanganate (dilute). This succeeded, and a very thick syrup was obtained which gave on analysis numbers corresponding to those calculated for the formula—



MEETINGS FOR THE WEEK.

- MONDAY, 21st.—Society of Arts, 8. (Cantor Lectures). "The Thermo-chemistry of the Bessemer Process," by Prof. W. N. Hartley, F.R.S.
- TUESDAY, 23rd.—Royal Institution, 3. "The Simplest Living Things," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
- WEDNESDAY, 23rd.—Society of Arts, 8. "The Preparation of Meat Extracts," by C. R. Valentine.
- THURSDAY, 24th.—Royal Institution, 3. "Recent Researches in Magnetism and Diamagnetism," by Prof. J. A. Fleming, M.A., D.Sc., F.R.S.
- FRIDAY, 25th.—Royal Institution, 9. "Canterbury Cathedral" by The Very Rev. The Dean of Canterbury, F.R.S. Physical, 5. "On the Circulation of the Residual Gaseous Matter in a Crookes Tube," by A. A. Campbell Swinton. "On some Improvements in the Roberts-Austen Recording Pyrometer, and Notes on Thermoelectric Pyrometers," by A. Stansfeld.
- SATURDAY, 26th.—Royal Institution, 3. "Portraits as Historical Documents," by Lionel Cust, M.A., F.S.A.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Gold Deposited on Iron.—Can anyone explain how gold is deposited on metallic iron? The facts of the case are as follows:—A piece of iron was taken from a pool of water (which contained copper in solution). After the copper was knocked off the iron the gold was exposed in very small plates. I have tested the gold with the usual tests.—B. H. B.

WILLESDEN DISTRICT COUNCIL.

SUPPLY OF SULPHATE OF ALUMINA.

THE WILLESDEN DISTRICT COUNCIL are prepared to receive Tenders for the Supply of SULPHATE OF ALUMINA for the Treatment of Sewage in the Brent District for the year ending March 31st, 1899.

All particulars may be obtained on and after Monday, March 14th, 1898, upon application to Mr. O. CLAUDE ROBSON, M.Inst.C.E., Engineer to the Council, Public Offices, Dyne Road, Kilburn, N.W. Tenders, endorsed "Sulphate of Alumina," to be delivered not later than 4 p.m. on Tuesday, March 22nd, 1898.

The Council do not bind themselves to accept the lowest or any tender.

(By Order)

Public Offices,
Dyne Road, Kilburn,
March 8th, 1898.

O. CLAUDE ROBSON,
Engineer to the Council.

COUNTY BOROUGH OF SALFORD. GAS DEPARTMENT.

TENDERS FOR SULPHURIC ACID.

THE GAS COMMITTEE invite Tenders for the Supply of about 1400 tons of SULPHURIC ACID, to be delivered during twelve months from date of acceptance of Tender. Full particulars may be obtained from the Gas Engineer, Gas Offices, Bloom Street, Salford. Sealed Tenders, endorsed "Tender for Acid," addressed to the Chairman of the Gas Committee, to be delivered to me not later than 3 p.m. on Thursday, March 24th, 1898.

(By Order)

Town Hall, Salford,
March 12th, 1898.

SAML. BROWN,
Town Clerk.

COUNTY BOROUGH OF WEST HAM.

THE COUNCIL of the Borough hereby invite APPLICATIONS from duly qualified persons for the following position on the Teaching Staff of the TECHNICAL INSTITUTE now in course of erection:—

HEAD OF THE CHEMICAL DEPARTMENT.

Full particulars of the duties attached to the post and of the salary to be paid may be obtained on application to the Principal of the Technical Institute, Town Hall, West Ham, E., before March 31st.

By order of the Council,

Town Hall, West Ham, E.,
March 16th, 1898.

FRED. E. HILLEARY,
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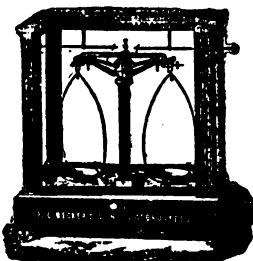


Fig. 11. No. 91.

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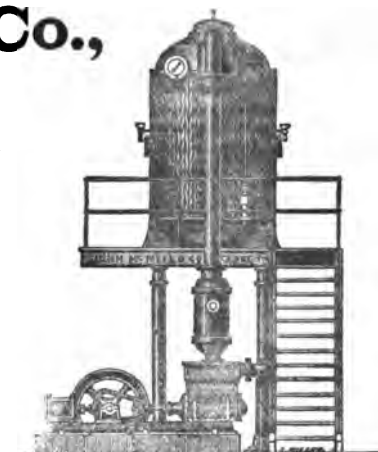
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CONTENTS.

ARTICLES:—	PAGE
Note on the Interaction of Cyanides with Thio-sulphates, by Leonard Dobbin, Ph.D.	131
Some Striking Lecture Experiments, with Indicators, by John Waddell	131
Separations from Chromic Acid, by Harry Brearley	131
Laboratory Notes, by H. Jervis	133
On the Occurrence and Extraction of Thorite, Monazite, and Zircon, by P. Truchot	134
The Simultaneous Volumetric Estimation of Sulphuric Acid and Lime in Waters, by Lucien Robin	135
The Action of Sulphuric Acid on Mercury, by J. R. Pitman	136
The Detection of Salicylic Acid in Foods, by F. A. Genth, Jun.	136
Diastatic Fungi and their Utilization, by Jokichi Takamine	137
Alkyl Bismuth Iodides and Bismuth Iodides of Vegetable Bases, by Albert B. Prescott	138
NOTICES OF BOOKS.—Spectrum Analysis	139
CORRESPONDENCE	139
CHEMICAL NOTICES FROM FOREIGN SOURCES	140
MISCELLANEOUS	141
MEETINGS FOR THE WEEK	142
NOTES AND QUERIES	142

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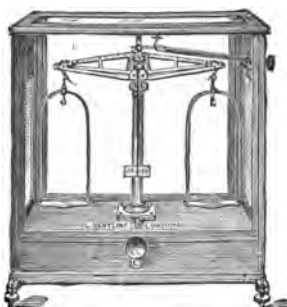
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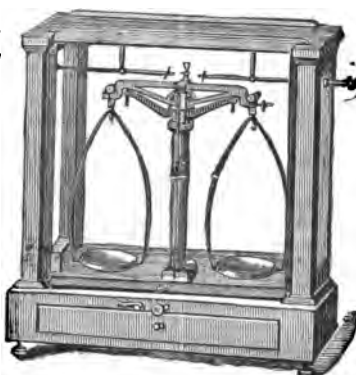


FIG. 3A.

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THE CHEMICAL NEWS.

VOL. LXXVII., No. 2000. 2

NOTE ON THE INTERACTION OF CYANIDES WITH THIOSULPHATES.

By LEONARD DOBBIN, Ph.D.

THE fact that an interesting change takes place when potassium cyanide and sodium thiosulphate are intimately mixed with each other in concentrated solutions was accidentally discovered, a short time ago, in the analytical laboratories here. The discovery was due to the circumstance that numerous students, in examining a certain mixture, designed as an exercise in qualitative analysis and containing these two salts along with other ingredients, altogether failed to detect the salt-radical of the thiosulphates, and were able to find traces only of that of the cyanides, whereas the indications of the presence of sulphites and of thiocyanates were strongly marked and unmistakable. A very short investigation of the matter showed that a simple change takes place whereby the thiosulphate parts with one-half of its sulphur and is converted into sulphite, while the cyanide is simultaneously converted into thiocyanate. The following equation represents the change that takes place when potassium salts are employed:—



One part by weight of purified potassium cyanide (quite free from thiocyanate) was ground up in a mortar with 3·2 parts by weight of crystallised potassium thiosulphate, and the resulting pasty mass was preserved in a closed tube. Immediately after mixing, a part of the mixture was dissolved in water, hydrochloric acid was added in slight excess, and then a drop of ferric chloride solution, but no thiocyanate reaction was observable, neither could any sulphite be detected. After twenty minutes the ferric chloride test was repeated with a fresh portion of the mixture, when quite distinct evidence of thiocyanate was obtained. On the following day the thiocyanate reaction was strongly marked, as was also that of sulphite, but cyanide and thiosulphate were evidently both present also, although the thiosulphate reaction was rather faint. After a week the presence of thiosulphate in small quantity was still recognisable on boiling some of the mixture with dilute hydrochloric acid, but the reaction of cyanide could no longer be obtained. This final test showed that the cyanide had been completely transformed into thiocyanate, and that the thiosulphate had been employed in slight excess from the beginning.

What seemed to be the most likely places in the literature have been examined with a view to finding any reference to this interaction as having been previously described, but so far entirely without success. It is therefore believed to be novel, and it is for this reason that it is now placed upon record.

Chemistry Department, University of Edinburgh,
March 14th, 1898.

SOME STRIKING LECTURE EXPERIMENTS, WITH INDICATORS.

By JOHN WADDELL.

In the course of an investigation on Indicators, the details of which are given in the March number of the *Journal of Physical Chemistry*, I have come upon some

striking experiments, which can be shown upon the large scale and are suitable for lecture purposes. They illustrate how the ordinary action of indicators used in alkalimetry may be completely masked by the addition of certain solvents.

The reactions are similar to that shown by cobalt chloride, which in alcoholic solution is blue, while the aqueous solution is pink. If water be added, in comparatively small quantity even, to the alcoholic solution, the pink colour appears, but the blue may be restored by the addition of ether. The phenomena are explained on the hypothesis that the dissociated salt of cobalt is pink and the undissociated salt blue, and that, in the presence of sufficient excess of alcohol or ether, the dissociation is diminished, giving the blue colour.

Cyanine in aqueous acid solution is colourless; but if a solution of cyanine be made in concentrated acetic acid, addition of alcohol or acetone turns it the blue colour which gives the substance its name. Water discharges the colour, which can be reproduced by further addition of alcohol or acetone, and the alternations of colour can be repeated almost indefinitely provided the reagents are not added in too great excess.

Methyl orange in acetic acid solution is red, but on addition of alcohol or acetone turns yellow (the same colour as in alkaline solution), but becomes red again on addition of water. The colours may be made to alternate as in the case of cyanine.

Paranitrophenol in ammoniacal solution is deep green; but if concentrated aqueous solution of ammonia is added to the paranitrophenol, acetone is capable of making the colour much lighter, and the further addition of ether discharges the colour entirely. Addition of alcohol, or more especially water, to the liquid thus obtained, produces an intense green.

It will be noticed that alcohol acts in this case like water, and not like acetone, the amount of dissociation in alcohol being sufficient to yield the colour of the ion.

Phenolphthalein in concentrated solution in acetone is scarcely coloured by addition of a considerable quantity of strong ammonia solution, but the further addition of water produces the very intense red characteristic of the alkaline solutions of this indicator.

If a solution of corallin in concentrated ammonia be made, addition of acetone will discharge the red colour, replacing it by a pale yellow, which turns back to red on addition of a little water, becoming once more yellow by addition of sufficient acetone.

The quantity of acetic acid or strong ammonia may be as large as 2 or 3 per cent of the total liquid.

It is to be specially noted that acetic acid and ammonia must not be replaced by hydrochloric acid and potash, as it is only with weak acids and bases that the phenomena can be readily observed.

Cornell University.

SEPARATIONS FROM CHROMIC ACID.

II. THE SEPARATION OF MANGANESE.

By HARRY BREARLEY.

ON adding potassium chromate to a neutral solution of a manganous salt there is no precipitate formed immediately, but on standing a dark brown precipitate is deposited. This precipitation is accelerated by heating, or if the operation be performed in a hot solution the precipitate forms at once. Various formulæ have been given to explain this reaction.

A circumstance in connection with manganese which does not obtain in treating iron or aluminium, is that the hydrate of a manganous salt exerts a strong reducing action, which is apparent from the well-known blackening of freshly precipitated $Mn(OH)_2$. Such hydrates formed

in a chromate solution would still more readily be oxidised, and cause the reduced chromic salt to be precipitated along with the manganese. Such, I imagine, is the cause of greater deficiencies than can be ascribed to the K_2CrO_4 and $MnCl_2$ reaction whenever the manganese salt is precipitated as hydrate.

Separation with Soda Hydrate.

In this and the following cases half a grm. of manganese (added as $MnCl_2$) is separated from 50 c.c. of a potassium chromate solution, after making acid and diluting to about half a litre. In no case was it possible to distinguish the precipitate as the white manganous hydrate, partly perhaps on account of the colour of the chromate, but more probably on account of its immediate oxidising effect.

It will be seen, from Table IV., that the usual increasing recovery attends the use of increasing amounts of alkali, and that the results are worse in hot than in cold solutions, though this latter may be due to the more energetic oxidising effect of the chromate in hot solutions, as well as to the increased vivacity of the reaction between the chromate and the manganous chloride. After filtering off a fraction of supernatant liquid the residue was poured into a strongly acidified solution of ferrous ammonium-sulphate, and titrated. Now, if the oxidation of the manganous hydrate had taken place solely at the expense of the chromate, the residue should have recorded an equivalent of K_2CrO_4 , which—with that in the fraction of its supernatant liquid—would make up the total amount of chromate added. In every case this amount was exceeded, so that, in spite of being suspended in an oxidising liquid, which it did not completely reduce, the manganous hydrate absorbed oxygen from the air also. The chromate value of this atmospheric oxidation of the cold precipitations are given in the table.

TABLE IV.

Excess 2N.NaHO.	K_2CrO_4 found in filtrate.		K_2CrO_4 value of atmospheric oxidation.
	Boiling solutions.	Cold solutions.	
1	3'4	16'4	14'3
10	7'7	21'1	15'7
20	10'0	29'0	17'2

In the hot solutions the atmospheric oxidation was much less.

This unpromising reaction has not, that I know of, been directly applied to the separation of manganous salts from chromic acid, but it seems to form a very serious drawback to an improved form of a well-known method of estimating chromium in steel, ferro-chromium, &c.

In 1877 (CHEMICAL NEWS, xxxv., 151) W. Galbraith described a method for estimating the chromium which was at that time being first introduced into steel. It consists briefly in dissolving the sample in dilute sulphuric acid, oxidising the Fe_2 to Fe_3 and the Cr_2O_3 to CrO_3 with permanganate, filtering off the manganese oxides, and titrating the chromic liquor with a ferrous salt and bichromate. With some slight modifications the method is still, at any rate in Sheffield, much used. In 1893 (*Journ. Iron and Steel Inst.*, i., 146) Galbraith deals with some objections which had been repeatedly urged against his method, but we are concerned only with the means he takes to free the precipitated manganese oxides from the chromic acid which it was said they retained. He might have reduced the needlessly large bulk of the oxides by adding less permanganate in excess, a procedure we had long before found to minimise the error; but he proposed instead to make the solution alkaline with soda hydrate before filtering, and then to wash the precipitate free from chromic acid. Mr. Galbraith's improved process, quoted *verbatim*, thus becomes:—"Dissolve in sulphuric acid (1 to 6), add permanganate, and boil till precipitate is black; then dilute and make alkaline with caustic soda,

filter, wash,* acidulate filtrate, and treat as before with the ferrous salt and potassium bichromate.

The method is illustrated by reducing and re-oxidising a standard solution of bichromate: this particular test I have repeated with fair results, but it must be pointed out that it is not fully representative of the conditions under which the method is intended to be applied. For instance, take a chrome steel; the dissolved sample consists of ferrous and chromic sulphates, and unless the equation—

$$2FeSO_4 + 4H_2SO_4 + 2KMnO_4 =$$

$$= Fe_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 4H_2O$$

wofully misrepresents the facts, there exists, after treating with permanganate, no inconsiderable quantity of manganous sulphate, which, on the subsequent addition of caustic soda, would be precipitated as the energetic reducing agent $Mn(HO)_2$, which, as is shown above, disputes the claim of the separation to be called qualitative even.

It is noticeable that the mere separation of the iron cannot be dismissed with the indefinite "make alkaline with caustic soda": there needs be more than a faint excess of soda.

I have made trials on artificial and actual examples of chrome steel, and have obtained results in keeping with the above explanations.

Mr. Galbraith further illustrates the improved method by results obtained on samples of ferro-chromium said to contain certain percentages, but he nowhere adopts the more satisfactory course of dissolving a pure carbon steel along with a known quantity of a chromium salt. In one case he treats 5 grms. of a sample said to contain 8 per cent Cr, and finds by his improved process that it contains 9.5 per cent. If the sample did really contain 9½ per cent, there was surely in the oxidation of the accompanying iron enough manganous sulphate formed to give the reducing action of the $Mn(HO)_2$ an opportunity of impressing itself on the observation. It may be recalled that larger excesses of alkali gave larger recoveries, and perhaps the same may be said for the permanganate; one can only suggest, in despair of offering a trustworthy explanation of Mr. Galbraith's results, that he uses these reagents in unusual excess: it may, at any rate, be set down that the process needs stating in more definite terms before it can be satisfactorily worked.

Separation with Bromine and Ammonia.

The advantage of this means of separating the manganese over the previous one consists in the presence of an oxidising agent ready to share the oxidation of the precipitated $Mn(HO)_2$ with the chromate. The separations are the better the more bromine there is present and the greater excess of ammonia there is added. Indeed an approximate accurate separation can be effected by using large quantities of these reagents, but the process is not to be compared with the following ones. Crookes ("Select Methods," p. 232) notices that when bromine is replaced by hydrogen peroxide or an hypochlorite, and the ammonia by soda, the manganese dioxide always contains chromium.

Separation with Soda Carbonate.

This reagent was added to boiling solutions of manganous chloride and potassium chromate; contrary to the behaviour of any other precipitant, an increased excess gave a decreased recovery. Soda carbonate was added also to a cold solution of the two salts and raised to boiling, and to a third set it was added; these last were filtered without any heating whatever. In the first case all the precipitates were dark coloured, but increasingly so as the alkali went up. In the second case the precipi-

* The precipitate would contain the excess of permanganate as manganous oxides, the oxidised $Mn(HO)_2$ precipitate, and the iron as ferric hydrate. When several grms. of a sample are taken these make up an enormous precipitate, which I am convinced cannot be thoroughly washed without great trouble.

tates were drab-coloured, and in the third case they might be described as creamy. The percentage recoveries are set forth in—

TABLE V.

Excess aN carb.	Boiling.	Cold- boiling.	Cold.
1	85.0	96.6	96.5
10	68.4	99.3	100.3
20	63.8	99.8	100.2

Sell's process ("Select Methods," p. 128) for estimating chromium consists in oxidising the chromic oxide with permanganate, adding alcohol and a slight excess of soda carbonate, filtering off the manganese, and titrating the filtrate. This method is illustrated by Table V. only in so far as the manganous salts formed by the oxidation of the chromic oxide are concerned, and, except with very large amounts of chromium, the error due to precipitating from hot solutions might be overlooked.

Separation with Soda Phosphate.

Gibbs's (CHEMICAL NEWS, xvii., 195) instructions for precipitating manganese as the double phosphate of manganese and ammonia are:—"Disodic ortho-phosphate is to be added in large excess above the quantity required to precipitate the manganese as ortho-phosphate. This precipitate is re-dissolved in HCl or H_2SO_4 , the solution heated to boiling-point, and ammonia added in excess. A white or semi-gelatinous precipitate is produced, which, on boiling or standing for some time, gradually becomes crystalline."

This process, applied to the separation of manganese from chromic acid, gives perfectly accurate results. If the soda phosphate be only in slight excess the results are a little low; if the ammonia, too, be only just in excess, the results are somewhat low; but an excess of 50 per cent phosphate and 20 c.c. normal ammonia, or more, always gives accurate results. This conclusion is based on a series of tests, in which each reagent was varied in turn: the above general statement may take the place of test separations which show no strong contrasts.

The crystallisation of the ammonia-manganese phosphate facilitates the estimation of the manganese; but if only the amount of chromic acid is sought for, it is unnecessary to wait for the precipitate to form the beautiful talcose scales, pearly in lustre and pale rose in colour; it is unnecessary even to re-dissolve the first formed precipitate: simply add soda phosphate, make alkaline, and, after settling, syphon a portion through a filter.

LABORATORY NOTES.

By H. JERVIS.

Reducing Action of Filter-papers.

THERE is no need to state this fact with respect to many oxidising agents, but with respect to bichromate solutions there is some difference of opinion. Thus Arnold ("Steel Works Analysis," p. 154), when estimating Cr by a modification of Galbraith's method, filters the hot chromate solution through a *pure* paper, although in a footnote he draws attention to the fact that thick filters of impure paper are liable to reduce a little CrO_3 to Cr_2O_3 , pure and ashless being apparently synonymous terms. I was recently made aware that one of the parties contending hotly over a difference of a few tenths per cent in a rich ferrochrome had filtered the hot chromate liquor through paper, unconscious of a possible reducing action.

To illustrate the point we have heated 25 c.c. of a solution of K_2CrO_4 (1 c.c. = 0.01 grm. Fe), diluted to 200 c.c. in a water-oven, then added a 12½ c.m. paper, and continued the digestion for half an hour. In one case the solution was made acid with 50 c.c. normal H_2SO_4 , and

in the other alkaline with 50 c.c. normal NaHO. A few cases in which the paper was added to a cold solution are also given:—

12½ c.m. Paper.	Ash.	C.c. K_2CrO_4 after digesting			
		Hot.		Cold.	
		Acid.	Alkali.	Acid.	Alkali.
English (Ford Mill, 428)	0.0048	23.7	24.8		
Schleicher & Schüll (No. 589)	0.0003	23.8	24.2	25.0	25.0
Thin German (S. & S.)	0.0028	24.2	24.6		
Ordinary " "	0.0028	23.6	24.6	25.0	25.0
Swedish (Munktell) ..	0.0005	24.6	24.9		
French White	0.0070	24.3	24.7		
French Grey	0.0139	22.5	24.0	24.4	24.7
White Blotting (Ford Mill, 428)	0.0095	22.9			

Bottle Labels.

The ordinary unprotected adhesive label does not long resist the attack of damp acid fumes which are always in the air of an active laboratory. Of the varnishes, balsams, &c., used to coat the surface, paraffin wax is about the most handy and serviceable.

Faraday recommended a mixture of Brunswick black with half its bulk of oil of turpentine; the mixture to be kept in a bottle whose stopper was perforated with a thick nibbed pen. A more tasty label of a similar sort may be made by grinding sealing-wax, first dry and then mixed with a little alcohol and ether in such proportion as to make a thick liquid. Such liquid is best applied to the bottle with a small stiff brush. If the bottle can be warmed it is feasible to write on the heated surface with the pointed end of a piece of sealing-wax. Such labels never "gang agley," as the most carefully varnished paper labels sometimes do.

Tests for Sulphurous Acid.

In reducing ferric solutions with sulphurous acid the sense of smell does not indicate the absence of SO_2 with sufficient delicacy; it is prone to be biased by colds in the head and other physiological derangements. To boil for an inordinate length of time after this test has given negative results, so as to make sure, is rather rule-o'-thumb. Because it is easy to apply, I propose, instead of a Bunsen valve and other like contrivances, that through the hole of the stopper which closes the mouth of the flask there be inserted the short end of a syphon-like tube; as soon as the evolution of SO_2 becomes faint, the other end drawn out is dipped into an acid solution of very dilute permanganate. A decolorisation which gives place to a brown precipitate is not to be regarded, but the complete clearing of the solution indicates SO_2 . This test, on account of large volume of vapour condensed, shows SO_2 after the sharpest noses have failed to detect it. The solution may be conveniently cooled and diluted by dipping the tip of the leg into cold water.

Interference of Manganous Salts with the Bichromate Estimation of Iron.

When a process fails, one naturally enquires after the author, but I am unable to find who is responsible for the suggestion that traces of iron may be estimated in the ignited Mn_2O_4 by dissolving in HCl, reducing, and titrating with bichromate. Such a procedure, however, I have seen practised—and, up to 1893, having unquestioned confidence in my superiors, I practised myself. I then had occasion to observe that the non-appearance of the blue colour was not a satisfactory proof of the absence of ferrous salts. The results of closer observation showed that a moderately strong solution of a manganous salt gives a brown colouration with ferricyanide, and that in presence of very small amounts of ferrous salts the blue colour may appear but momentarily and then give place to the brown colouration, or not appear at all. Thus, with as much Mn_2O_4 as one obtains from 1½ grms. of a 20 per cent spiegel, it was found after dissolving in HCl

and diluting to 100 c.c. (many times greater bulk than I have seen employed) that ferrous sulphate equivalent to 0.003 grm. Fe_2O_3 could escape detection. In very dilute solutions the Mn does not give a brown colour with ferricyanide, and hence the use of a very dilute solution of ferricyanide—used, say, $\frac{1}{2}$ c.c. at a time instead of in drops—enables us to avoid the interference, but this device somewhat impairs the delicacy of the reaction. As some much-used processes include titrations of diluted ferrous salt in presence of manganese, it was thought worthy to notice this source of error, although the particular operation in connection with which it was observed may have grown obsolete. When using the sulphocyanide colour test, the presence of manganese introduces a slight negative error of no practical importance. It may be interesting to notice that the presence of manganese chloride heightens the accuracy of a proportional contrast; that is, solutions of equal volume, containing respectively 0.0001 and 0.0003 grm. Fe, are more nearly alike in tint than when the latter is diluted three times in presence of MnCl_2 than without it.

ON THE OCCURRENCE AND EXTRACTION OF THORITE, MONAZITE, AND ZIRCON.*

By P. TRUCHOT.

THE recent progress made in gas-lighting depends on the principle of producing a complete combustion of the hydrocarburetted materials and using the great amount of heat thus produced for rendering incandescent certain oxides selected and arranged in the most suitable manner.

Many savants have been working on this idea for some years, but without good results. It was not until a few years ago that Auer von Welsbach, inspired by the work of earlier chemists, succeeded in producing in an elegant and practical manner a method of applying the then rare oxides to the purpose of incandescent gas lighting.

The present Auer mantles are composed of about 99 per cent of oxide of thorium and 1 per cent of oxide of cerium; while the former ones, dating from 1883, were formed, above all, of oxide of zirconium and oxide of lanthanum.

These rare oxides, the basis of the incandescence, are very difficult to obtain, and we propose in this article to study the principal minerals now being used for their production, namely, thorite, monazite, and zircon. We shall give their composition as well as their principal sources.

I.—Thorite.

Thorite is a hydrated silicate of thorium; its formula is $\text{SiO}_4\text{Th} \cdot 2\text{H}_2\text{O}$. It contains a small amount of oxides of iron, manganese, calcium, magnesium, lead, tin, and a fairly noticeable amount of oxide of uranium.

In nature it is found in the form of dodecahedral crystals with two tetrahedral faces, unequally developed, or of an hexagonal appearance.

It is in colour darkish brown or orange (one variety being named *orangite*); it has a resinous lustre and a conchoidal fracture. In thin flakes it is translucent or transparent.

It has been found in syenite at Lowö, near Brewik, in Norway; but the only deposits really known and worked for the manufacture of mantles are those of Langesundfjord, between Arendal and Christiania.

Table I. shows the average composition of two samples of thorite and one of orangite from these Norwegian deposits.

TABLE I.—Average Composition of the Minerals of the Thorite Group.

	Orangite.	Thorite.	Thorite.
Silica (SiO_2)	17.32	18.98	17.04
Oxide of thorium (ThO_2) ..	71.65	57.91	50.06
Oxide of uranium (U_2O_3) ..	1.13	1.58	9.78
Oxide of lead (PbO)	0.88	0.80	1.67
Sesquioxide of iron (Fe_2O_3) .	0.59	5.79	7.60
Alumina (Al_2O_3)	0.17	0.06	—
Oxide of cerium (Ce_2O_3) ..	—	—	1.39
Lime (CaO)	1.59	2.58	1.99
Magnesia (MgO)	traces	0.36	0.28
Alkalis	0.47	0.24	—
Water (H_2O)	0.14	9.50	9.46
Density	5.19	4.80	4.38

II.—Monazite and Monazite Sands.

Monazite is essentially an anhydrous phosphate of cerium, lanthanum, and didymium; its formula may be represented by $(\text{CeLaDi})_2\text{O}_3\text{P}_2\text{O}_5$ (Penfield). It always contains—and this is what makes it valuable from our present point of view—variable quantities of oxide of thorium, which may exist in the form of silicate, as in thorite and orangite, or as a phosphate forming a double salt with phosphate of cerium, or even as an amorphous mixture.

Monazite contains a small quantity of zirconia, alumina, magnesia, lime, oxides of iron, manganese, tin, and lead, besides small quantities of titanate acid and fluorine. It crystallises in clinorhombic prisms, either translucent or transparent, of a yellowish, reddish yellow, brownish, or greenish colour, with a resinous lustre. The brownish or yellowish fragments appear to be the richest in oxide of thorium.

This mineral which, as recently as three years ago, was considered to be extremely rare, has become relatively common—thanks to the discovery of such enormous deposits of monazite sands—that even the incandescent gas-lighting industry may very reasonably have disappeared before one thousandth part of it has been used.

Monazite has been found in large quantities in the United States, Canada, and Brazil; it has also been noticed in England (in Cornwall). It has been met with in Sweden, at Holma, Karafoet, and Johannisberg; in Norway, at Arendal, Hitteröe, Noterö, and Midbö; in Russia, in the Ilmen Mountains and in the placers of the river Sanarka; in France it has been found at Puy, near Saint Christophe; and, finally, a mine has just been discovered at Ryfylke, in Norway.

In each of these deposits the monazite is one of the secondary constituents of the eruptive rocks, granite and gneiss. It has been found in a large number of similar rocks, and a more careful study will certainly reveal the fact, in a few years, that all granites and gneiss contain it in more or less quantities. All the most important deposits (the United States, Brazil, Siberia, &c.) thus come from the disaggregation of the primitive granitoid rocks under the influence of variations of temperature, erosion, &c., and are, as a rule, found localised in beds, or at the source of small rivers, and on the sea-shore.

The monazite originating from the destruction of these rocks is naturally mixed with a number of other minerals, which form with it what is known commercially as the *monazite sands*.

The principal minerals present are—Zircon (which is always present, and often in larger quantities than the monazite), then *xenotime*, *fergusonite*, *sphene*, *rutile*, *garnet*, *brookite*, *ilmenite*, *apatite*, *magnetite*, and sometimes *corundum*, *colombite*, *samarskite*, *gadolinite*, and *orthite*.

The most important deposits of monazite sands actually known can be divided into two principal groups:—(1) Those in the United States and Canada; (2) those of Brazil, Columbia, and the Argentine Republic.

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Deposits in the United States and Canada.

The deposits in the United States are mostly in North Carolina, but some are in South Carolina and Virginia, while it has quite recently been discovered in the State of Idaho.

1. *North and South Carolina.*—The area covered by these deposits is about 1600 square miles, spreading over the counties of Burke, McDowell, Rutherford, Cleveland, and Polk (North Carolina), in a part of the county of Spartanburg (South Carolina), and in the county of Amelia (Virginia). The principal deposits of this region are in the Silver, North Muddy, and South Muddy streams, the Catawba river, the Second Broad River, and the First Broad River (Rutherford, Cleveland, and Spartanburg). All these rivers have their sources in the mountains in the south, not far from the Blue Mountains. The rock is composed of granite, biotite, gneiss, diorite, and hornblende, cut almost at right-angles by a parallel system of small veins of auriferous quartz. The greater part of this region has been worked for gold.

It was in 1879 that, for the first time, Mr. W. Hidden found workable quantities of monazite. These are, as a rule, found at the sources of the above-named rivers mixed with gravel and ordinary sand. The thickness of the deposits of these monazite sands is from 0.3 to 0.6 metre, while the breadth of the streams is often 3.6 metres. The proportion of raw monazite present is extremely variable, oscillating between merely traces and 1 or 2 per cent. Naturally the richest deposits are nearer the source of the streams—the heavier minerals, particularly monazite, being concentrated there.

The monazite from these deposits, as we learn from a memoir by Mr. Nitze, is extracted by washing the sand and the gravel in sluices—exactly in the same manner as for alluvial gold. The sluice boxes are about 2.4 metres long by about 0.5 metre in width, and about 0.5 metre in depth. Two men work the sluice box; one shovels the sand on to a perforated screen fixed at the upper extremity of the apparatus, while the other keeps the material inside in constant movement, either with a fork or a perforated spade, in such a manner as to eliminate the less dense sands. These boxes are cleaned up at the end of every day's work; the washed and concentrated monazite being collected and dried.

If the sand obtained contains magnetic iron, the latter can be removed by means of a strong magnet, but the greater part of the heavier minerals cannot be eliminated—there is always zirconium, rutile, brookite, corundum, garnet, &c., remaining. Commercial monazite sand thus prepared is not pure, but is only a mixture of heavy minerals in which monazite predominates. A sand containing from 65 to 70 per cent of monazite is considered to be of very good quality. A good daily return varies from 18 to 32 kilos. of monazite sand per sluice box.

As there are but few regular mining undertakings existing in this region, as a rule each claim holder works his own claim, and sells the monazite to the local buyers—or, as often happens, exchanges it for produce.

As a matter of fact, with but few exceptions, the monazite deposits in the river beds have been almost entirely exploited; it follows, then, that the operations are now directed to the deposits of gravel in the adjacent valleys.

These deposits are worked by small shafts about 0.75 of a metre square, which enables the sand to be extracted and put in the sluice box arranged at the mouth. The upper layers are generally rejected, except when they are found to contain a little monazite. The shafts are arranged in parallel lines, similar to the method adopted in gold placer workings. At the Blanton mine, on the Hickory river, two miles north-east of Shelby, in Cleveland county, the deposit is from 90 to 120 metres in width, and has been partly worked for a distance of a quarter of a mile along the bank of the river. The upper

layer has a thickness of 0.9 to 1.0 metre, and the layer of monazite sand 0.3 to 0.9 metre.

In the county of Spartanburg, in South Carolina, the extraction and concentration by washing and deposition are carried out in a much more perfect manner, and, although the raw sands contain a much larger proportion of garnet, rutile, and titanite iron, the commercial product is obtained much more economically in the form of monazite sands of different grades.

Two sluice-boxes, one placed above the other, are used for the washings. The sand is shovelled on to a perforated plate fixed at the upper end of the first box, and a subsequent washing gives a sand of great purity, often containing 85 per cent of monazite. The tailings from this washing (the lighter particles) are discharged directly into the lower box, where they are washed over again and give a sand of the second grade.

These sands are almost always further treated by machines similar to those used for winnowing wheat. In this manner a black sand is separated consisting of quartz and very fine fragments of monazite, which constitutes the final tailings. It is impossible to further wash these sands without a loss of monazite, or to separate any more of the garnet, rutile, or titanite iron which must always be present, even in the best samples.

If the monazite gravel contains gold this latter will be found with the monazite, and this is by no means rare, since in most auriferous placers monazite has long been found in the residue thrown on one side from the washings of the gold-bearing sands.

(To be continued).

THE SIMULTANEOUS VOLUMETRIC ESTIMATION OF SULPHURIC ACID AND LIME IN WATERS.

By LUCIEN ROBIN.

I HAVE attempted the rapid estimation of the sulphuric acid and the lime in waters; these two bodies being of much importance, as much from the point of view of potability as for use in boilers.

We take 100 c.c. of the water to be analysed and place it in a specially shaped flask having an expansion in the neck above the 100 c.c. mark, add $\frac{1}{2}$ c.c. or 1 c.c. of the alkaline solution used in the estimation of the ammonia by Nessler's solution,* then boil gently for about five minutes; cool under the tap, bring up to 100 c.c. again, shake thoroughly, and filter exactly 50 c.c. The filter and the funnel are placed over the special flask.

Estimation of the Sulphuric Acid.—We use the well-known method of precipitation by a known quantity of chloride of barium, then titrating by chromate to measure the excess. We prefer to work in the following manner:—To the 50 c.c. of the filtered solution we add two drops of litmus and acidulate with five or ten drops of pure hydrochloric acid, and boil for three or four minutes to drive off the carbonic acid. We then add a few drops of ammonia to neutralise the acid, and, without taking away the flame, pour in exactly 10 c.c. of a N/20 solution of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, then withdraw the heat and allow to stand for fifteen minutes. After this time, boil and add 10 c.c. of a N/20 solution of chromate.† After a few minutes' boiling, cool under the tap and filter. The flask must be washed out twice with enough water to fill the filter each time.

* Pure crystallised carbonate of soda, 100 grms.; caustic soda in alcohol, 50 grms.; distilled water, 300.

† We weigh exactly 3.690 grms. of bichromate of potassium and dissolve it in water, saturate the solution with pure ammonia, and make up to 1 litre. It is necessary to make quite certain that 10 c.c. of this solution corresponds exactly to 10 c.c. of the solution of barium—but this is not a difficult matter,

To the filtrate we add 20 c.c. of a solution of ferrous sulphate (10 grms. of the salt and 20 c.c. of sulphuric acid per litre), then titrate the excess of the ferrous salt with decinormal permanganate of potash—let us suppose that we have used 3·3 c.c. of this latter. On the one hand, we take 5 c.c. of the chromate solution, to which we add 20 c.c. of the ferrous solution, and by means of permanganate we estimate the quantity of the ferrous salt unoxidised by the chromate, say, 16 c.c.

On the other hand, we estimate the quantity of permanganate necessary to oxidise 20 c.c. of the ferrous solution, say, 19·5 c.c. Thus, 19·5 c.c. - 16 c.c. = 3·5 c.c., corresponding to the iron oxidised. This understood, let us suppose that in an estimation we have used 3·3 c.c. of permanganate, we should then say 19·5 c.c. - 3·3 c.c. = 16·2 c.c., which would represent the quantity of permanganate corresponding to the ferrous salt oxidised by the chromate in excess, from which we deduce—

$$x = \frac{5 \times 16.2}{3.5}$$

x being the number of c.c. of chromate in excess, from whence $x \times 0.068 = \text{CaOSO}_3$ per litre of water; in fact, the number of c.c. of chromate in excess represents that of the solution of barium which was used to precipitate the sulphuric acid.

Comparative Estimations in M.grms. per Litre.

By weight.	Volumetrically.	Oxygen absorbed.*
11'0	9'0	1'0
70'0	72'2	4'1
349'0	343'0	4'0
396'4	398'0	5'8
641'3	639'2	6'3

* We give the oxygen absorbed by the organic matter to show that this latter has very little influence on the results.

Estimation of the Lime.—The filter which was used for the filtration in the estimation of the sulphuric acid is moistened with 5 c.c. of half-strength hydrochloric acid, and washed by filling up the filter twice with warm water. This is then made slightly ammoniacal, and we then add 10 c.c. of a solution of decinormal oxalate of ammonia. Make up to 100 c.c. and, after shaking, let stand for at least half an hour. After this time we can do one of the two following things:—(1) Estimate in an aliquot part—50 c.c., for example—the excess of oxalic acid after having filtered it and made it slightly acid with a little pure nitric acid; or (2) estimate the oxalic acid in the precipitate after having filtered and then washed twice with ammoniacal water, and finally with warm water. The filter placed over the flask where the precipitation was made is washed three times with 5 c.c. of pure dilute warm nitric acid. After the solution of the oxalate, we wash twice with boiling water, filling the filter each time, and in the liquid we estimate the oxalic acid after having heated it to about 60°. The number of c.c. of permanganate used $\times 0.028$ give the total quantity of lime present in 1 litre of water.

Comparative Estimations in M.grms. per Litre.

By weight.	Volumetrically.
9'6	8'5
80'0	77'0
226'0	222'0
247'0	241'0

If the water contains much, it suffices to augment the quantity of the reagents used. If the *special* flask is not used, it is as well to boil in a flask gauged to 200 c.c. and make up to this amount, then filter 100 c.c. instead of 50 c.c. for estimating the sulphuric acid.

By this method, one can estimate several samples of water, both for sulphuric acid and lime, in less than an hour.—*Journal de Pharmacie et de Chimie*, Series 6, vol. vii., No. 6.

THE ACTION OF SULPHURIC ACID ON MERCURY.

By J. R. PITMAN.

THE following statement is made by Messrs. Baskerville and Miller, in a previous number of the *Journal of the American Chemical Society* (xix., 874):—"A qualitative experiment showed that mercury decomposed concentrated sulphuric acid at the ordinary atmospheric temperature, about 20° C."

As a large amount of my work consists in the use of nitrometers, this statement was of considerable interest to me; however, believing their conclusions to be erroneous, the following simple tests were tried:—Apparatus used, a Lunge nitrometer, with separate reading burette; the temperature at all times was about 22° C.; the amount of mercury was from eight to ten times that of the acid (by volume).

First experiment: 30 c.c. of concentrated sulphuric acid was run into the generating bulb, and allowed to stand for forty-eight hours, being shaken at intervals. I was unable to get any gas at all under these circumstances, and there was apparently no reaction between the mercury and the sulphuric acid. Thinking that possibly the presence of air might have some effect upon the reaction, I next measured a certain quantity (about 50 c.c.) of rather damp mercury: this was run into the generating bulb and 30 c.c. of sulphuric acid as well; after shaking at intervals for twenty-four hours, the air was re-measured and found to have lost four-tenths c.c.: this loss was probably due to the presence of considerable moisture in the air when first measured; as a check this same air was conducted (thoroughly dried from its contact with the sulphuric acid) from the reading burette into another generating bulb, drawing in 30 c.c. of concentrated sulphuric acid, and shaking again, as before, for about twenty-four hours, with a result of a loss of less than 0.05 c.c., which is an error that might occur in any test.

In order to try the effect of the preponderance of sulphuric acid, 1 part of mercury to 70 by volume of concentrated sulphuric acid was taken (sp. gr. 1.84), introduced into a flask, and shaken violently for some time; no mercuric sulphate was formed, nor was there appearance of any other reaction; this was at a temperature of 25.5° C. From these experiments it is apparent that there is no reaction between mercury and sulphuric acid at ordinary temperature, and if Messrs. Baskerville and Miller found a reaction as they state, it must have been by means of some different method.—*Journal of the American Chemical Society*, vol. xx., No. 2.

THE DETECTION OF SALICYLIC ACID IN FOODS. (PRELIMINARY NOTE).

By F. A. GENTH, Jun.

IN the examination of foods for salicylic acids to so-called distillation method, suggested by G. Krause and fully described by Mr. McElroy, is the one most frequently used.

The method is as follows:—The mass is pulped with phosphoric acid in a mortar, water added if necessary, and after some time strained through a cloth. About 50 to 75 c.c. of the liquid is then subjected to distillation and successive portions of 5 c.c. each of the distillate collected and tested with ferric chloride. If salicylic acid has been used as a preservative, it will be distilled with the water vapour, and give a violet colouration when ferric chloride is added. In the first portions of the distillate no reactions may be obtained, and in cases where only very small amounts of salicylic acid are present in the article under examination, the reaction in the last portion of the

distillate would not be very marked. The methyl ester reaction, which is likewise recommended as a test for salicylic acid, cannot always be obtained when the amount present is small.

No other substances have been known to yield distillates giving similar colourations, or any reactions interfering with that for salicylic acid under the above circumstances.

The writer has recently had occasion to examine a series of preserves, such as jellies, jams, catsup, &c., for preservatives. Among the samples submitted were a number obtained through the courtesy of Food Inspector H. J. Hackett, and were known to be free from salicylic acid or other preservatives. In the examination of these latter samples according to the above-described method, a colour reaction similar to that given by salicylic acid was found in all cases. When tested with ferric chloride the first portions of the distillate showed little if any colouration, and only as the distillation continued did it become more marked. In many cases the distillation process was not continued to within the last 10 c.c., but was stopped with over 25 c.c. still remaining in the flask. The colouration in dilute solutions was violet, of about the shade and colour of similar ferric salicylate solutions; in more concentrated solutions the colour was much redder in the case of the distillate from the pure food, and was found to effectually hide the presence of small amounts of salicylic acid, when added.

The nature of the distillates and the examination of food-stuffs for salicylic acid will be further investigated.—*Journal of the Franklin Institute*, cxliv., No. 3.

DIASTATIC FUNGI AND THEIR UTILISATION

By JOKICHI TAKAMINE.

UP to the present time the germination of cereals has been the only source of diastase of any practical importance known in America and Europe. It is true that there is diastase of animal origin, such as ptyalin and pancreatic diastase, but their sources are limited and their potency unstable. Therefore they are comparatively of less importance than the vegetable diastase, which has an inexhaustible supply of raw materials of uniform power. In Japan, and some other Asiatic countries, certain kinds of fungi are used in the production of diastase. The fungus that is in use in Japan is called *Moyashi*, which was named by Ahlburg *Eurotium Oryzae*. It belongs to the genus *Aspergillus*, and is distinguished from ordinary fungus by its remarkable power of generating diastase during its growth. It is a perfectly harmless plant, as proven by the fact that it has been used in Japan for several centuries in the manufacture of the various daily beverages. Further study has shown that a good many other fungi have, to a more or less degree, a similar property, and naturally, therefore, the selection of the species which has the strongest diastase generating power becomes of technical importance. This selection is determined by the culture, multiplication of each species, preparation of diastatic substance, and the comparison of the diastatic power of different species under similar conditions. Such selected species are then subjected to cultivation for the purpose of obtaining their matured spores in the production of diastatic substances. For this purpose suitable materials, such as rice, hominy, ground corn, or wheat bran, are thoroughly steamed so as to sterilise the mass, as well as to gelatinise the starch, and the product is supplied with an artificial fertiliser to give the plant the sufficient amount of nutriment for its complete maturity. On to this mass selected culture is sown, after which it is put into an incubator of proper temperature and humidity. Inside of twenty-four hours the fungus growth will become visible, and at the end of six or seven days the growth will reach its maturity, pre-

senting a rich, velvety appearance of colour, varying from reddish to dark green, according to the species of plant used. This product is carefully dried and preserved. The matured spores of the plant may be separated from the mass by shaking or sifting, and then can be preserved indefinitely. The product thus obtained is called *Taka-Moyashi*, and is used as the seed spore in the manufacture of diastatic substances.

To produce diastatic substances for commercial purposes, wheat bran is first moistened with water and then thoroughly steamed. After the mass is cooled down below 40° C., a small quantity of *Taka-Moyashi* is added and thoroughly mixed. The mass is then taken into a growing-room similar to that of a malt-floor, and spread in a layer varying from 1 to 2 inches in thickness. The temperature of the room is kept at about 25° C., and the humidity at above 80 per cent. Inside of twenty-four hours the fungus shows its growth, and the diastatic strength of the mass will steadily increase as the growth advances, and it will be found that within from forty to fifty hours the diastatic power reaches its maximum, after which the mass is taken out of the growing-room and cooled down to ordinary temperature to check the further growth of the plant. The mass thus obtained is called *Taka-Koji*, and can be used as it is in the green state, or it can be dried for preservation. As the diastase generated in *Taka-Koji* is readily soluble in water, the mass may be percolated with cold water and the extract thus obtained can be used as a diastatic agent, or, for the same purpose, it may be mixed with the extract of ungerminated cereals, which have the singular property of augmenting the diastatic power of *Taka-Koji*.

This extract, for the purpose of preservation, may be evaporated under a vacuum to a thick syrupy condition. In this condition its diastatic power is from eight to ten times stronger than that of malt extract of similar consistency. It can be applied to all such industries as the manufacture of alcohol, beer, vinegar, &c., where the diastase performs the important function of converting starch into sugars.

The aqueous extract of *Taka-Koji* can still be further purified by precipitating the diastatic principle of the extract by the addition of alcohol. For this purpose an extract containing about 20 per cent of solid matter is mixed with four to five times its own volume of strong alcohol. By this means the diastase, together with some other albumenoids, is precipitated, while the sugars and other impurities remain in solution. The precipitate is now separated from the mother-liquor by decantation and centrifugal force; it is then pressed and air-dried. The product thus obtained is called *Taka-Diastase*. It is a yellowish-white odourless powder, possessing a nutty taste. It is readily soluble in water, yet it is non-hygroscopic. It is perfectly stable in its diastatic power. It converts in ten minutes over 100 times its own weight of starch, according to the modified Junk's test. It has remarkable starch liquefying property besides starch-saccharifying property, the former being three or four times stronger than that of purified malt. It is strong enough for all practical purposes. It can be, however, further purified to wonderful strength by re-precipitation or otherwise.

The applications of *Taka-Diastase* are varied and extensive. From the remarkable stability of its diastatic power it can be used as a standard of comparison in the determination of the diastatic power of other substances. Its use as a remedy for amylaceous dyspepsia is of no mean importance.

Considering the fact that more than two-thirds of our food consists of starch substances, such as potato, bread, pudding, &c., and also that the diastase of the saliva has to perform the principal function in the digestion of starchy food, and that the saliva is subjected to loss and deterioration of its diastatic power from various causes, such as smoking, drinking, chewing, and rapid eating, it is not to be wondered at that two-thirds of the dyspepsia

is of a starchy origin, and therefore it is apparent that some kind of strong diastatic substance is required to supply the deficiency of the diastatic power in the system of the digestive organs.

While our knowledge is very limited of the quantitative estimation of the diastase daily generated, and contained in the saliva and pancreatic juices, as far as my investigation goes the quantity of diastase secreted in the saliva daily is very considerable. It amounts to from 5 to 8 grms. of Taka-Diastase, or about $\frac{1}{4}$ pound of the best malt extract. While investigation in the way of the production of diastase from a fungus growth is still in its infancy, yet that which we already know on this subject seems to indicate that this has opened an entirely new field for the economic production of diastatic ferments. I firmly believe that this field will, in the future, supersede in every respect the old known source, namely, the germination of cereals.—*American Journal of Pharmacy*, vol. lxx., No. 3.

ALKYL BISMUTH IODIDES AND BISMUTH IODIDES OF VEGETABLE BASES.*

By ALBERT B. PRESCOTT.

THE common alkyl ammonium iodides, with solutions of the bismuth salts, give bright-coloured precipitates. As formed by quaternary methyl or ethyl ammonium iodides, the colour is orange-yellow in most cases, usually lighter when obtained with bismuth chloride, and darker when obtained with bismuth nitrate. When fully formed by excess of the organic iodide, in bismuth solution not strongly acid, the precipitation is amorphous and so nearly complete that when the filtrate from a test-tube portion is evaporated to dryness and the residue ignited and treated with solvent acid, hydrogen sulphide fails to blacken the liquid. Strong mineral acids slowly decompose these coloured precipitates, liberating iodine.

What has been known as Dragendorff's reagent for alkaloids is a potassium-bismuth iodide, prepared by dissolving precipitated bismuth iodide in a concentrated solution of potassium iodide acidulated with hydrochloric acid, and known as giving reddish coloured precipitates in solution of the salts of the alkaloids. On trial with pyridine salts, a corresponding precipitate was obtained, dark orange-red and voluminous. Kraut (*Ann. Chem. (Liebig)*, ccx., 310—327) has reported the piperidine compound, to which all these are analogous.

These organic bismuth iodides are not perfectly proof against decomposition by much water; they are sparingly soluble in ethyl or amyl alcohol, insoluble in glacial acetic acid, in ethyl ether, in chloroform, and in benzene.

The tetramethyl ammonium-bismuth iodide crystallises from hydrochloric acid, that of sp. gr. 1.19 diluted with an equal measure of water. Also from potassium iodide solution acidulated with hydrochloric acid. The pyridine and the alkaloid bismuth iodides crystallise from alcohol. In all these cases the crystals are clearly hexagonal and easily obtained.

Both the amorphous and crystalline forms are stable in the air. A sample of tetramethyl ammonium bismuth iodide remained constant in weight at 130°; atropine bismuth iodide melts, but at 98° C. holds constant weight.

Reducing agents, as potassium thiosulphate, do not alter these bismuth iodides. Tetramethyl ammonium bismuth iodides, precipitated from 10 per cent solutions both of the organic iodide and bismuth iodide, washed with hydrochloric acidulated water, then with pure water till washings gave no residue, then with alcohol, and

lastly with ether, and dried at 110°, gave figures as follows:—

Analyses I. and II. were of the same preparation; III. and IV., from other preparations made at different times.

	I.	II.	III.	IV. $N_5(CH_3)_{11}HBiI_4$
Iodine ..	58.71	58.29	60.79	59.22
Bismuth ..	27.08	—	—	27.07
Carbon ..	8.69	8.68	8.58	8.82
Hydrogen ..	2.16	2.12	2.30	2.37
Nitrogen ..	2.81	2.73	—	2.81

The pyridine bismuth iodide, prepared from a pyridine salt by Dragendorff's reagent and crystallised from alcohol, on elementary analysis gave figures as follows:—

	I.	II.	III. $(C_5H_5N)_2(HBiI_4)$
Iodine ..	63.98	62.84	62.24
Bismuth ..	23.36	23.72	—
			63.59
			23.18

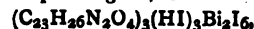
Kraut (*Ann. Chem. (Liebig)*, ccx., 310—327) found analogous composition for piperidine bismuth iodide.

The corresponding atropine bismuth iodide gave results as follows:—

	I.	II.	III. $(C_{17}H_{23}NO_3)_2(HBiI_4)$
Iodine ..	46.99	47.03	46.51
Bismuth ..	18.82	18.67	18.53
Carbon ..	23.22	23.62	23.69
Hydrogen ..	2.87	3.02	2.94
Oxygen ..	8.10	7.66	8.33

The carbon is too low for the theory, so that the figures approach to those of $(C_{17}H_{23}NO_3)(HBiI_4)$.

The brucine compound gave, for—



of iodine 41.10 and 40.88 per cent, against 41.56 by calculation from the formula.

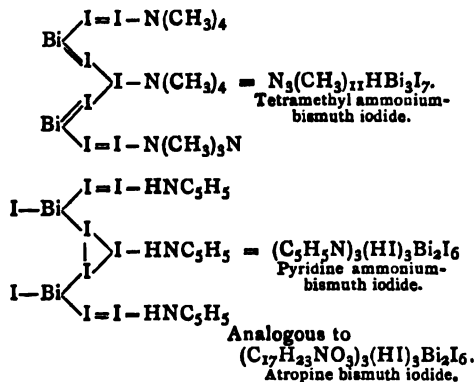
The corresponding strychnine salt gave, for iodine, 44.02 and 44.65 per cent against 44.48 by calculation from the formula.

In the results of the work I have done upon the perhalides and double halides of nitrogen bases in the last two or three years, everything goes to support the theory that two or more halogen atoms link to each other with an (uneven) valence of more than one, so as to connect one group of atoms with another. Iodine, especially among the halogens, serves as a binding element in the coupling of other elements with each other, as well as in massing its own atoms together in a heavy periodide, as a swarm of bees hang upon the bough of a tree. In the double iodide structure, where iodine links one base to another base, serving between positive and positive, with iodine not in excess of the "normal" number of its atoms, potassium thiosulphate will not take iodine out of the compound. In the periodide structure where iodine links a base to additive iodine, serving between a positive and a negative group, potassium thiosulphate promptly removes all the additive iodine, leaving a normal single iodide. These generalisations apply to the dipyridine alkyl iodides reported with determinations of molecular weight by Mr. Flintermann and myself in 1895 (*Journ. Am. Chem. Soc.*, xviii., 28). Also to the various monopyridine alkyl normal iodides (Prescott and Baer, 1896, *Ibid.*, xviii., 247) and to the numerous periodides (Prescott and Trowbridge, 1895, *Ibid.*, xvii., 859; P. F. Trowbridge, 1897, *Ibid.*, xix., 322; Trowbridge and Diehl, 1897, *Ibid.*, xix., 558). In the compounds of additive iodine, as in double iodides, the results of analyses are in most cases consistent with an uneven valence of iodine, indeed with its trivalence. But there are a very few periodides well determined by Mr. Trowbridge as monopyridine compounds, in which, in our present knowledge, an even numerical valence of iodine is indicated.

The bismuth iodides of nitrogen bases reported in this paper—both those of fatty alkyls, on the one hand; and

* Read before the American Pharmaceutical Association, in the report of a special committee, at Minnetonka Lake, August, 1897, and to be published in the *Association Proceedings* for this year. From the *Journal of the American Chemical Society*, xx., No. 2.

those, on the other hand, of pyridine and pyridine-derived alkaloids—all evidently conform in their analytical content to the regular iodine-linking structure—the structure common both to double iodides and periodides, as shown in the following proposed constitutional formula. In the case of the quaternary ammonium bismuth compounds, with the prevailing bismuth characteristic of losing halogen in presence of water, the wash-water being found tinged with iodine, each bismuth atom is directly bound to only two atoms of iodine, while in the pyridine-formed compounds the bismuth atom is bound to three atoms of iodine in each instance, bismuth and iodine valencies being always the same. Again, the fatty ammonium compounds, less stable as they are, show a variation from the quaternary to the tertiary base type, in one of the three nitrogen basal groups of the molecule. This doubtless comes about by reaction of water to form methyl alcohol as a by-product, leaving hydrogen in place of methyl in the main product.



The alkaloidal bismuth iodides are not quantitatively uniform enough to be entirely satisfactory for alkaloidal assay, but are more stable and uniform than the alkaloid mercuric iodides formed by Mayer's reagent. On the other hand, they are more bulky, less easy to gather in a compact mass, less manageable in filtration. On the whole, so far as found, Dragendorff's reagent gives no general advantage over that of Mayer, though I am well aware how unsatisfactory the latter has been found in the hands of analysts.

For the execution of the work upon tetramethyl ammonium bismuth iodide, I am wholly indebted to Mr. H. E. Brown; for that upon the bismuth iodides of pyridine and the alkaloids, to Mr. O. C. Diehl. A further study of the reactions of the halides of bismuth upon representative organic bases is now left to Mr. Brown.

NOTICES OF BOOKS.

Spectrum Analysis, by John Landauer. Authorised English Edition. By J. BISHOP TINGLE. First Edition, first thousand. New York: John Wiley and Sons. London: Chapman and Hall, Ltd. 1898. Pp. 239. 8vo., 134 figures.

DR. LANDAUER's excellent treatise first appeared as an article contributed to Fehling-Hell's "Neues Handwörterbuch der Chemie," and was published as a separate book at the request of a number of competent authorities. Dr. Tingle's translation now makes it available for English readers. Among the features of this very practical book are full bibliographical references to authorities suitable to the historical treatment of the subject. The older measurements, to secure uniformity, have been re-

calculated to accord with Rowland's system of wave-lengths.

A summary of the chapters will give a good idea of the contents of the book:—I. Historical Introduction. II. Physical Properties of Light. III. Spectroscopes. IV. Spectroscopic Instruments for Special Purposes. V. Spectroscopic Adjuncts. VI. Emission Spectra. VII. Spectra of the Elements. VIII. Absorption Spectra. IX. The Solar Spectrum. X. Other Celestial Bodies.

In Chapter VII. the wave-lengths of the spectrum lines of the several chemical elements are given very fully, in alphabetical order of the elements.

The book is notable for the very concise and clear style of the author, which has been admirably preserved in the translation; some paragraphs, as those in the last chapter, are very brief, yet they indicate the essential facts necessary for a student to know, without a superfluous word. For students having a knowledge of the elements of physics the book will be of great value.

There are two useful indexes, one for authors and one for subjects, in accordance with the custom of certain editors, though one index in a single alphabet is far to be preferred. The illustrations are well chosen, and the paper and type worthy of the firm that has done so much to advance science by placing valuable books in the hands of teachers and pupils.

H. C. B.

CORRESPONDENCE.

THE INTERACTION OF MAGNESIUM AND SOLUTION OF SULPHATE OF COPPER.

To the Editor of the Chemical News.

SIR,—In a communication to the Chemical Society, on the 3rd inst., Dr. Divers disputes the suggestion of Prof. Clowes, that it is the combined action of magnesium and copper sulphate in water that causes the evolution of hydrogen gas when these substances are brought together. He lays especial stress on copper sulphate being an acid salt, and capable of being decomposed into sulphuric acid and a basic sulphate.

I have made lately a large number of experiments on the action of magnesium on metallic salts, and some of my results point distinctly in favour of Clowes and Caven's theory. For instance, if magnesium is placed in a cold strong solution of either sodium chloride, calcium chloride, or calcium nitrate, a copious evolution of hydrogen takes place, and the solutions become distinctly alkaline to litmus or phenolphthalein. A white solid residue is left in each case, consisting in the case of calcium chloride at least of both Mg and Ca oxides. With the calcium salts sufficient Mg is taken into solution to give a distinct precipitate with the ordinary reagents; I have not been able to obtain a precipitate from the solution of NaCl, which simply shows that MgO is no more soluble in NaCl than in pure water.

These are all neutral salts, and yet their solutions are decomposed by magnesium in the cold. Ammonium salts, such as the chloride and nitrate, give a similar reaction, but, as is well known, ammoniac hydrate alone has the same action on this metal.—I am, &c.,

E. G. BRYANT.

King's School, Pontefract,
March 20, 1898.

On Hydrocinnamide.—Marcel Delepine.—Hydrocinnamide crystallises with half a molecule of water, and has the fundamental properties of the glyoxalidines; it is a base, giving salts undecomposable by acids; the body $\text{C}_{15}\text{H}_{17}\text{N}_3$ does not exist. The author thinks the name *cinnamine* would be more appropriate.—*Comptes Rendus*, cxixvi., No. 9.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 8, February 21, 1898.

Chemical Actions brought about by the Electric Discharge. (Methods).—M. Berthelot.—The author has paid particular attention to the fixation of nitrogen by the organic compounds under these conditions, and his experiments are now complete on 120 systems. The general arrangement of the experiments was to place the substance to be operated upon in a narrow tube of convenient shape, in such a position that it must play the part of a dielectric, and be incessantly traversed by the discharges of a high tension apparatus such as a Ruhmkorff coil, fitted with a Leyden jar and a Marcel Deprez interrupter, giving many hundreds of interruptions a second. The reactions studied were between gases, liquids, and solids, the conditions being varied in the case of each one. Carbides of hydrogen and the compounds containing little oxygen gave first a little acetylene, but this soon disappeared. Compounds containing much hydrogen, such as formene, when in the presence of nitrogen, first gave ammonia, but this also soon disappeared, and it often happened that large volumes of gas were first produced, which were eventually re-absorbed by reason of fresh reactions. In general the nitrogen fixed under the prolonged influence of the electric discharge appears to exist as an ammoniacal derivative, such as an amide or amine, but especially as a polyamine.

Chemical Action exercised by the Electric Discharge on Organic Compounds. (Gaseous Systems; Carbides of Hydrogen and Nitrogen).—M. Berthelot.—A long paper, in which the author gives the results of a large number of experiments by the above-mentioned method on formene, CH_4 ; hydride of ethylene, C_2H_6 ; ethylene, C_2H_4 ; acetylene, C_2H_2 ; propylene, C_3H_6 ; trimethylene, C_3H_8 ; and acetylene, C_2H_2 .

The Derivatives of Cinchonine.—E. Grimaux.—As a starting point the author took the bromised compound obtained by treating the raw product from the oxidation of cinchonine, or of quinine by bromine, and obtained $\text{C}_9\text{H}_{14}\text{BrNO}_2\cdot\text{HBr}$. By treating this with picric acid he obtained a picrate of bromomexzoquinene; this can be dissolved in strong potash on the water-bath, and on cooling a deposit of crystalline plates is obtained. He also obtained acetyl-mexzoquinene and oxymexzoquinene.

On a Combination of Phosphoric Anhydride and Benzene.—H. Giran.—Already inserted.

Ktypeite, a New Form of Carbonate of Lime, different from Calcite and Arragonite.—A. Lacroix.—This new mineral constitutes exclusively the pisolites from the thermal springs of Carlsbad and Hammam-Mescoutine, often described as having been formed from arragonite. When heated in a glass tube these crystals first of all decrepitate at a dull red heat and then detonate with violence, often shattering the glass. Ktypeite evidently exists under conditions of extreme tension, for after having been tapped with a hammer, though not sufficiently hard as to break the crystals, but only to disintegrate them, it can be heated to redness without detonating. This extreme state of tension is also shown by its action with parallel polarised light. Its density is from 2.58 to 2.70, that of calcite being 2.71.

No. 9, February 28, 1898.

Chemical Actions of the Electric Discharge. (Oxides of Carbon and Nitrogen; Gaseous Systems).—M. Berthelot.—In the present memoir the author commences with the binary compounds, such as carbonic acid

and carbonic oxide; he then goes on to the study of the ternary compounds, alcohols and ethers, aldehyds and acids. The paper, however, is a very long one and not suitable for abstraction.

Properties and Crystallisation of Anhydrous Sulphide of Barium.—A. Mourlot.—Sulphide of barium was prepared by means of the electric furnace; first the pure amorphous sulphide was produced by the action of sulphuretted hydrogen on the carbonate at a red heat, taking care to substitute hydrogen for the former gas towards end of the experiment; the density of this sulphide, taken in benzene at 15° , is 4.30 when powdered and 3.95 in large fragments. Analyses give its composition as:—

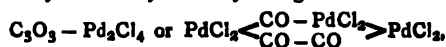
		Theory.
Ba.. ..	81.5	81.09
S	18.03	18.91

Sulphate of barium was then intimately mixed with the necessary amount of carbon, and after heating strongly in the electric furnace for four minutes, a mass was obtained which showed a distinct crystalline formation in the interior. Analyses showed it to be:—

		Theory.
Ba	81.20	81.29
S	18.65	18.30
		18.91

Its density is practically the same as the former product. It crystallises in white crystals, colourless when seen in thin layers, but black when thicker, owing to the presence of carbon. They have two very distinct cleavage planes, at right angles, and have no action on polarised light. The hardness of crystallised sulphide of barium is not great, as it will not scratch glass. It is readily attacked by fluorine, with incandescence, as well as by chlorate of potassium and oxide of lead in the same manner. Phosphoric anhydride and oxychloride of phosphorus reduce it with incandescence, forming a reddish body.

Action of Carbonic Oxide on Palladous Chloride.—E. Fink.—Palladous chloride is heated to about 260° in a glass tube, and a current of dry carbonic oxide passed over it; after some time the cool part of the tube becomes coated with a reddish substance near the furnace and yellow in the cooler parts of the tube; this substance can only contain palladium, chlorine, and carbon, and it is shown that it consists of a mixture in variable proportions. The analysis of the yellow crystals gives the formula—



decomposable by water; heated to 263° it loses carbonic oxide and forms a new compound corresponding to the formula COPdCl_2 or $\text{CO}=\text{PdCl}_2$.

On an Oxyptomaine.—O. de Coninck.—The manner of formation of the new compound described in this note, together with its properties and its reactions, show it to be an oxycollidine, $\text{C}_8\text{H}_{10}(\text{OH})\text{N}$, a higher homologue of the oxypyridines already described by the same author; the name *collidone* is proposed. This one appears to be the first known.

Zeitschrift für Analytische Chemie.
Vol. xxxvii., No. 1.

Estimation of Carbon and Hydrogen by Combustion in Vacuo.—K. A. H. Möerner.—The principle of this method is the same as that of Klingeman. The substance (0.08—0.12 grm.) is mixed with powdered copper oxide and asbestos fibres, and placed in a tube closed at one end; the tube is nearly filled up with granular copper oxide. The tube is then placed in the channel of a water-bath, and connected with a mercury-pump. During the pumping, the water surrounding the tube is heated to boiling-point, so that any water inside the tube may vapourise and the pumping be made easier. When the tube is exhausted, the other end is closed, the tube placed in the furnace, and the combustion proceeds in the ordinary

way. The products of combustion are collected in a eudiometer and analysed.

A New Method of Estimating Grape Sugar and Milk Sugar.—E. Riegler.—The sugar solution is treated with Fehling's solution; the copper is estimated, before and after the reduction, by iodine, according to de Haen's method. Before milk sugars can be estimated, all albuminous substances must be removed by asapol. To do this 15 c.c. of asapol and citric acid solution are put, together with 10 c.c. of milk solution, into a 100 c.c. flask, which is then filled up to the mark with water. The liquid is well shaken, warmed to 60°, and filtered. The sugar can then be estimated by taking 20 c.c. of this sugar solution free from albuminous matter, and 20 c.c. of Fehling's solution, and boiling for about six minutes. In the experiment described the reduced copper amounted to about 100 m.grms., and 1 m.grm. Cu corresponds to 0.725 m.grm. of milk sugar.

Research on the Composition of Acid Thorium Oxalates.—Carl Glaser.—A thorium earth was brought into solution by treating it with H_2SO_4 , and after filtration the thorium was precipitated by oxalic acid. After careful washing, the oxalate was boiled with excess of strong ammonium oxalate solution, until it dissolved; after cooling it was filtered, and the double salt decomposed by acid. To determine what was the influence of the temperature on the reaction, three experiments were made, which gave nearly coincident results (51 per cent ThO_2). On leaving the precipitate for a long time in contact with the acid, there was a slightly higher yield (52.57 per cent ThO_2). The estimation of the oxalic acid and water was conducted in the ordinary way, and the formula found to be either $\text{Th}_2\text{H}_4(\text{C}_2\text{O}_4)_{12} + 25\text{H}_2\text{O}$ or $\text{Th}_2\text{H}_2(\text{C}_2\text{O}_4)_5 + 9\text{H}_2\text{O}$.

A Small Spirit-flask.—Jos. Loczka.—The flask is drawn and described.

An Evaporating Funnel.—D. S. Bosnjakovic.—This vessel can be used both as an evaporating basin and a filter, and so it prevents the necessity of transferring a precipitate from an evaporating basin to the filter. Two diagrams are given showing the position of the funnel in its two uses.

Estimation of Chromium in Chrome-iron Ore.—H. Fresenius and H. Bayerlein.—A weighed quantity of finely-powdered ore is digested in a 300 c.c. flask with 50 c.c. of concentrated HCl, at first in the cold, and as soon as the evolution of gas ceases it is gently warmed, until a very slight insoluble residue remains. The liquid and the residue are placed in a basin and evaporated to dryness. Silicates are then removed with HCl and water, and the liquid is filtered. The dark coloured residue is fused in a silver crucible with Na_2O_2 , and then treated with water and the yellow solution filtered. There remains on the filter-paper a black residue, insoluble in HNO_3 . The solution is treated with HCl to precipitate any silver, and the filtrate concentrated by evaporation. The residue is now treated with a little HCl and water, and again excess of Na_2O_2 is added, by which some iron precipitate is obtained free from chromium. To free the solution from excess of acid, it is placed in a 500 c.c. flask, together with the solution obtained from the first fusion with Na_2O_2 ; excess of Na_2O_2 is added, and the solution shaken and warmed, until the evolved gas ceases to come off. It is filtered, and the iron precipitate obtained, after washing, is dissolved in HCl. The solution is again concentrated by evaporation, added to the residue, and once again treated with Na_2O_2 . By this method the iron precipitate is always free from chromium. To separate the chromium from its strong alkaline solution it must be concentrated, and then carefully neutralised with HCl, and H_2O_2 added until the slight blue colour of the solution is changed to green on warming. The solution is now boiled, the H_2O_2 decomposing, and the liquid evaporated to dryness. The residue is treated with HCl, and the chromium will come down with ammonia free from silicates. If the chromium oxide obtained is not a pure product, it can be fused with

soda and saltpetre, treated with water, and the aqueous solution decomposed with ammonia and ammonium carbonate.

Journal de Pharmacie et de Chimie,
Series 6, vol. vii., No. 4.

Action of Iodine on Antipyrine. Application to the Estimation of Antipyrine and of Iodine. — J. Bougault. — The author has made a certain number of experiments, with the object of determining the conditions under which the absorption of iodine by antipyrine takes place, and he arrives at the conclusion that the reaction which takes place in alcoholic solution is such that one molecule of antipyrine used corresponds very exactly with one molecule of iodine. If to 20 c.c. of a solution of antipyrine (1 grm. per 100 c.c. of alcohol we add 20 c.c. of a solution of bichloride of mercury (2.5 grms. per 100 c.c. of alcohol), and then, drop by drop, a solution of iodine (at 1.351 grms. per 100 c.c. of alcohol) until the appearance of a persistent faint yellow colour, showing an excess of iodine, the number of c.c. of iodine used, multiplied by 5, will give the number of grms. per cent of pure antipyrine in the sample under examination. Inversely the amount of iodine present in any sample can easily be estimated.

The Estimation of the Phospho-glycerates. — MM. Adrian and Trillat.—When we add sulphuric acid of a known strength to a cold aqueous solution of a neutral phospho-glycerate of an alkaline earth, and use heliantine as an indicator, the indication appears from the moment that a half molecule of acid has been added for one molecule of the neutral salt. This fact shows that sulphuric acid transforms the neutral phospho-glycerate into an acid salt, and that the latter is not decomposed by the sulphuric acid,—at any rate not in the cold.

Volumetric Estimation of Combined Sulphuric Acid.—F. Telle.—As a modification of previous processes, the author proposes to estimate the excess of chromate by titrating, by means of hyposulphate, the iodine which is displaced, in hydrochloric solution. Two solutions of equal strengths, of chloride of barium and bichromate of potassium, are used. The sulphuric acid is precipitated by the chloride of barium after acidulating with HCl. The excess of baryta is then precipitated by the bichromate of potassium in slightly alkaline solution, and, finally, the excess of chromate is estimated by the iodine displaced by the hyposulphite.

MISCELLANEOUS.

Royal Institution.—The following are the Lecture Arrangements after Easter:—Mr. T. C. Gotch, two lectures on "Phases of Art, Past and Present"; Dr. S. R. Gardiner, four lectures on "The Historical Development of Europe"; Professor S. H. Butcher, two lectures on "Literary Criticism in Greece"; The Rev. Canon Ainger, three lectures on "Some Leaders in the Poetic Revival of 1760 to 1820—Cowper, Burns, and Scott"; The Right Hon. Lord Rayleigh, F.R.S., three lectures on "Natural Philosophy"; Dr. E. E. Klein, two lectures on "Modern Methods and their Achievements in Bacteriology"; Sir Walter Parratt, four lectures on "Programme Music" (with musical illustrations); Mr. J. A. Thomson, two lectures on "The Biology of Spring"; Dr. R. Caton, two lectures on "The Temples and Ritual of Asklepios at Epidaurus and Athens." The Friday evening meetings of the members will be resumed on April 22nd, when Mr. W. H. M. Christie, the Astronomer Royal, will deliver a discourse on "The Recent Eclipse." Succeeding discourses will probably be given by Prof. A. Gray, Mr. E. A. Minchin, Prof. W. A. Tilden, The Right Hon. D. H. Madden, Lieut.-General The Hon. Sir A. Clarke, Prof. W. M. Flinders Petrie, The Right Hon. Lord Rayleigh, and other gentlemen.

Chemical Actions of the Electric Discharge. (Alcohols and Derived Ethers in the presence of Nitrogen.—M. Berthelot.—In this paper the author describes the reactions of the electric discharge on the methylic, ethylic, normal- and iso-propylic and allylic alcohols, the reactions being carried to their extreme limits; he also includes experiments on various phenols as well as others with alcoholic compounds formed by dehydration, such as a simple ether, glycolic ether or pseudoxide of ethylene, and the dialcoholic ethers. The results of these experiments are all given at great length.—*Comptes Rendus*, cxixvi., No. 9.

Corundum Deposits in Eastern Ontario.—Until about thirty years ago the only sources of corundum were a few river washings in India and elsewhere, where it occurred in scattered crystals. In 1869 it was found by Mr. W. P. Thompson in Northern Georgia, U.S.A.; but late in 1896 Mr. Ferrier, of the Geological Survey, made a discovery of corundum in the northern part of Carlow Township, Hastings County, Canada. He made careful examination of the occurrence of the ore, and, taking back with him to Ottawa a lot of samples, he reported his discovery to Dr. Geo. M. Dawson, Director of the Survey. Dr. Dawson, in turn, reported it to the Bureau of Mines here, and as the mineral was on Crown lands the Commissioner of the Lands Department withdrew from sale a large block in the vicinity pending a more careful exploration. A quantity of the ore was taken out under his instructions, and samples were distributed to prospectors and others in the eastern part of the province. The result was that several other discoveries were soon reported, and early last summer Mr. W. G. Miller, of the Kingston School of Mining, was employed to make a thorough exploration of the country. He spent ten weeks in the northern part of Hastings and the southern part of Renfrew, and succeeded in tracing the corundum belt for a distance of 30 miles across country from Carlow in the west to Sebastopol in the east. The mineral was found in place in seven different townships,—Carlow, Bangor, Raglan, Radcliffe, Brudenell, Lynedoch, and Sebastopol,—over an area of about 200 square miles. There are indications, however, that the belt extends far north-eastward into Renfrew, as float boulders have been discovered at several points beyond the eastern end of the explored belt. If it turns out, as appears almost certain, that the corundum can be smelted economically for the production of aluminium, this alone will give it a value second in importance to none other in the province. A sample of the ore containing tin, as well as numerous samples of corundum, have been placed in the collection of minerals in the Parliament Buildings, Canada, where they are attracting considerable attention.

MEETINGS FOR THE WEEK.

- MONDAY, 28th.**—Society of Arts, 8. (Cantor Lectures). "The Thermo-chemistry of the Bessemer Process," by Prof. W. N. Hartley, F.R.S.
- TUESDAY, 29th.**—Royal Institution, 3. "The Simplest Living Thing," by Prof. E. Ray Lankester, M.A., LL.D., F.R.S.
- Society of Arts, 8. "English Art in Illuminated MSS.," by Sir Edward Maunde Thompson, K.C.B., D.C.L.
- WEDNESDAY, 30th.**—Society of Arts, 8. "Telegraphy Across Space," by Professor Silvanus P. Thompson, F.R.S.
- Microscopical, 8. Exhibition of Mounted Rotifers, by C. F. Rousselet.
- THURSDAY, 31st.**—Royal Institution, 3. "Recent Researches in Magnetism and Diamagnetism," by Prof. J. A. Fleming, M.A., D.Sc., F.R.S.
- Chemical, 3. Annual General Meeting.
- Society of Arts, 4.30. "The Earthquake in Assam," by Henry Lutman-Johnson, I.C.S.
- FRIDAY, April 1st.**—Royal Institution, 9. "Liquid Air as an Analytic Agent," by Professor Dewar, F.R.S.
- SATURDAY, 2nd.**—Royal Institution, 3. "Portraits as Historical Documents," by Lionel Cust, M.A., F.S.A.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Indicators.—Can any reader tell me of another indicator which may be used in place of ferricyanide in the estimation of phosphorus by uranium acetate? I wish to apply this method to estimating phosphorus in pig-iron and steel, and of course the iron will render ferricyanide useless.—V. G. J.

Products of the Distillation of Wood.—I am desirous of procuring some information in regard to manufacture and chemical analysis of the products resulting from the destructive distillation of wood, such as wood alcohol, acetate of lime, &c. In other words, information pertaining to running a chemical plant, manufacturing wood alcohol and making charcoal to the best advantage. Some good book on the above, if there is one, is what I would like. Any information on this subject will be gratefully received and appreciated.—E. E. J.

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- WANTED**—Any Vols. or Nos. of the *Journal of the Society of Chem. Industry*, 1882-86, *The Journal of the Chemical Society*, 1849-80, *The Analyst*, *Journal of Iron and Steel Inst.*, 1869-80, *Proc. of the Royal & Phys. Soc. of Edin.*, Gmelin's "Chemistry," vl. 19 (Index). Graham's "Physical Researches," and any Standard Literature.
- WILLIAM F. CLAY, Bookseller, 18, TEVIOT PLACE, EDINBURGH.

COUNTY BOROUGH of WEST HAM.

The COUNCIL of the Borough hereby invite APPLICATIONS from duly qualified persons for the following position on the Teaching Staff of the TECHNICAL INSTITUTE now in course of erection:—

HEAD OF THE CHEMICAL DEPARTMENT.

Full particulars of the duties attached to the post and of the salary to be paid may be obtained on application to the Principal of the Technical Institute, Town Hall, West Ham, E., before March 31st.

By order of the Council,

FRED. E. HILLEARY,
Town Clerk.

Town Hall, West Ham, E.,
March 16th, 1898.

JAMES HICKISSON, otherwise HICHISSON (trading as JOHN BOND), deceased.

NOTICE pursuant to Statute 22 and 23 Victoria, chapter 35, is hereby given, that all persons having any claim or demand against the Estate of the late JAMES HICKISSON, otherwise HICHISSON, of Erleigh, Gordon Road, Bournemouth, in the county of Southampton, formerly of Wilton, Drummond Road, Bournemouth, aforesaid, who carried on business at 75, Southgate Road, London, N., as a Marking Ink and Rubber Stamp Manufacturer, and who died on the 29th September, 1897, and whose Will and Codicils were proved by John James Rorie Friend and Duncan Watts, two of the Executors, on the 15th December, 1897, in the Principal Registry, are required to send particulars in writing of such claims or demands to the undersigned before the 23rd day of April, 1898, after which date the Executors will distribute the assets among the persons entitled, having regard only to the claims of which they shall then have had notice.

Dated this 23rd March, 1898.

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Superintendent of the Laboratory:
Dr. ALEXANDER SCOTT, M.A., D.Sc.

This Laboratory, which has been founded by Dr. LUDWIG MOND, F.R.S., as a Memorial of Davy and Faraday for the purpose of promoting original research in Pure and Physical Chemistry, is now open.

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All persons desiring to be admitted as workers, must send evidence of scientific training, qualification, and previous experience in original research, along with a statement of the nature of the investigation they propose to undertake.

The terms during which the Laboratory is open are the following—

MICHAELMAS TERM—First Monday in October to Saturday nearest to the 15th of December.

LENT TERM—Monday nearest to the 15th of January to the second Saturday in April.

EASTER TERM—First Monday in May to the fourth Saturday in July.

Candidates must apply for admission during the course of the preceding Term.

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CONTENTS.

ARTICLES:—	PAGE
On the Connection between the Electrical Properties and the Chemical Composition of Different Kinds of Glass, by Prof. Andrew Gray, LL.D., F.R.S., and Prof. J. J. Dobbie, M.A., F.R.S.	143
On the Occurrence and Extraction of Thorite, Monazite, and Zircon, by P. Truchot	145
A New Method of Fractionation of the Yttria Earths, by C. Urbain	147
PROCEEDINGS OF SOCIETIES—	
CHEMICAL SOCIETY.—Announcement by the Council—The Reduction of Bromic Acid and the Law of Mass Action—The Action of Ferric Chloride on the Ethereal Salts of Ketone Acids—Note on the Volatility of Sulphur—Cannabinol	148
PHYSICAL SOCIETY.—The Circulation of Gaseous Matter in a Crookes Tube—Thermo-electric Pyrometers	150
CHEMICAL NOTICES FROM FOREIGN SOURCES	151
MISCELLANEOUS	153
MEETINGS FOR THE WEEK	153

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THE CHEMICAL NEWS.

VOL. LXXVII., No. 2001.

ON THE CONNECTION BETWEEN THE ELECTRICAL PROPERTIES AND THE CHEMICAL COMPOSITION OF DIFFERENT KINDS OF GLASS.*

By Professor ANDREW GRAY, LL.D., F.R.S.,
and
Professor J. J. DOBBIE, M.A., D.Sc.

THE experiments and results described in the following paper are a first instalment of work we have undertaken with a view to determining, if possible, the circumstances which affect the conductivity and specific inductive capacity of glass. It appeared from some experiments which were carried out by Professor T. Gray and ourselves some years ago (*Roy. Soc. Proc.*, No. 231, 1884) that it might be of interest to have a number of glasses specially made up with a view to testing some of the conclusions then arrived at.

A result previously obtained by Professor T. Gray had shown that potash and soda-lime glasses have a higher conductivity than flint glasses; this result had also been arrived at by Dr. Hopkinson. In particular it seemed desirable to ascertain whether by increasing the amount of lead oxide and diminishing the amount of soda, the conductivity would go on diminishing. We have experienced great difficulty in getting glasses made according to our own specifications. We endeavoured to make the glasses ourselves, and several experiments were made accordingly, both in the laboratories here and at the Ogwen Tile Works, where a large furnace had been erected for the construction of tiles from slate dust. Some success was achieved, but it was found impossible, without the expenditure of far more time than could be spared, to obtain the glasses in a condition suitable for the experiments we wished to carry out.

Through the kindness, however, of Messrs. Schott and Co., of Jena, and of Messrs. Powell and Sons, Whitefriars, London, we have recently obtained a number of specimens of glass all richer in lead than the specimens formerly available, and, further, in some cases, practically free from soda. We have also had made to order by Messrs. Schott specimens of their own glass, used, we believe, chiefly in the construction of thermometers, as well as of a barium crown glass, which have not hitherto, so far as we are aware, been experimented with.

Determination of Conductivity.—The method of experimenting followed was practically the same as that described in the paper already referred to, but its nature may perhaps here be indicated.

Owing to the large percentage of lead oxide in some of the glasses prepared for us by Messrs. Schott, it was found impossible to blow them into flasks, and they were therefore cast into plates; the arrangements therefore required some modification for their case.

The specimens which were in the form of flasks were filled up with mercury to the bottom of the stem (which in most cases was about 8 or 9 inches long), and the flask thus filled was sunk in a bath also containing mercury, so that the mercury was at the same level inside and outside. One terminal of a circuit containing a battery of about thirty secondary cells and a very sensitive galvanometer was connected to the mercury within the flask by a wire passing down the neck, while the other terminal was connected to the mercury in the bath.

The galvanometer was the instrument formerly used

and described in *Roy. Soc. Proc.*, vol. xxxvi., p. 287. It was carefully insulated, as was also the reversing key, and all necessary precautions were taken to make sure that the current passing through the galvanometer was that passing through the walls of the flask between the mercury coatings. Thus it was always verified that no deflection took place when the wire was withdrawn from the flask and placed round the outside of the neck. The bath could be heated to any temperature required in the experiments.

The conductivity was calculated from the dimensions of the flask and the thickness of its walls, as described in the former paper.

In the capacity measurements the plate or flask, as the case might be, was supported as described above. A quadrant electrometer was kept connected to the plates of one of Lord Kelvin's air leydens. This was charged with twelve secondary cells, and therefore to a difference of potential of about 24 volts. After the battery had been removed a reading was taken of the electrometer deflection, and then the specimen was connected for a very short interval of time as a condenser in parallel with the leyden.

This connection was made by means of a myrograph pendulum which, when freed, swung over a considerable arc to a catch which prevented it from returning. At its lowest point a metal piece projecting below the bob touched the top of a tongue projecting upwards from a hinge at its lower end, and leaning against the point of an adjustable screw. The connection between the two condensers thus only endured while the three pieces, the screw, tongue, and bob, were in contact. This was only the interval of time required for a pulse of flexure to travel about a centimetre in a bar of steel about half a centimetre thick and about a centimetre broad. The interval was reckoned as at most about $1/30,000$ of a second.

The plates were originally rather over a quarter of an inch thick, and after some observations of capacity had been made on some of them, and it had appeared that their resistance was too great to be measurable, they were cut down on a turning-table used for cutting slates, to a thickness of about 3 m.m.; they were then fixed on a bed of pitch, and ground down by hand to a thickness of about 0.24 c.m. They were polished and properly cleaned, and then covered on both sides with a dense and thoroughly adherent coating of silver. This was cut away for a space of about half an inch round the edges. Great care was taken to remove every trace of silver, and to make the edge thoroughly clean.

While being experimented on the glass plate was laid with one coating of silver resting on a plate of copper at the bottom of an iron bath. Another plate of copper was laid on the upper coating of silver, and kept down with a weight, and the connections of the battery circuit, described above, were made with the copper plates. The iron bath was placed within a larger bath partly filled with sand, so that the temperature could be raised by heating the outer bath from below.

The results of the experiments are exhibited in the accompanying table. We have there given the density, specific resistance, specific inductive capacity, and chemical composition of each specimen experimented on.

The resistance was taken after five minutes' electrification in each case. The "Jena" glass (XXIV. of the table), in both resistance and capacity experiments, showed very considerable polarisation effects, which were a very long time in disappearing, though the conducting coatings of the flask were kept short-circuited. The polarisation of the lead glass made by Messrs. Powell was also considerable.

On the other hand, it is very remarkable that the barium glasses XXIII., in the capacity experiments, showed little or no sign of polarisation effects; and we propose to make some further investigations of the physical properties of this glass.

* A Paper read before the Royal Society, February 17th, 1898.

CHEMICAL COMPOSITION.

No.	Description of glass.	Density.	Specific resistance (ohms).	Specific inductive capacity.	Silica.	Lead oxide.	Ferric monoxide and alumina.	Manganese monoxide and arsenious oxide.	Potassium oxide.	Sodium oxide.	Total percentage.	Remarks.			
XXI.	Lead potash glass made by Messrs. Powell and Sons, London.	3.495	Too high to measure, certainly above $18,000 \times 10^{10}$ at 130°C .	7.966 at 15°C . 7.630 at 120°C .	50.5	42.14	0.41	—	6.93	trace	99.98	Absorption effects in electrical experiments very marked.			
XXII.	Lead potash glass made by Messrs. Schott and Co., Jena.	3.591	Too high to measure, certainly above $35,000 \times 10^{10}$ at all temperatures to 135°C .	7.991 at 14°C .	44.5	46.6	trace	0.4	8.0	0.3	100.0	Absorption effect slight.			
XXIII.	Barium glass made by Messrs. Schott and Co., Jena.	3.565	Too high to measure, certainly above $59,000 \times 10^{10}$ at all temperatures to 140°C .	8.5. No variation with temperature. No absorption effect.	Silica.	Boron tri-oxide.	Alumina.	Manganese monoxide and arsenious oxide.	Zinc oxide.	Barium oxide.	Magnesia.	Potassium oxide.	Sodium oxide.	This glass showed no trace of polarisation.	
XXIV.	Zinc soda 'Jena' glass, made by Messrs. Schott and Co.*	3.493	596.5×10^{10} at 43°C . 0.200×10^{10} at 140°C .	7.54 at 15°C . Conduction too great at high temperatures.	67.6	8.0	trace	0.4	9.0	—	5.0	—	10.0	100.0	Considerable absorption effects.

Dimensions of Specimens.

Internal radius.	External "	Effective surface	XXI. Flask.			XXII. Plate.			XXIII. Plate.			XXIV. Flask.		
			Length of silvered surface	Breadth of ditto.	Area.	Length of silvered surface	Breadth of ditto.	Area.	Length of silvered surface	Breadth of ditto.	Area.	Effective area.	Mean thickness	
..	..	..	12.81 c.m.	..	..	12.81 c.m.	..	..	18.8 c.m.	..	..	209.2 sq. c.m.	..	0.935 c.m.
..	..	..	11.90 "	..	..	11.90 "	..	..	14.31 "	..	..	269.028 sq. c.m.	..	0.235 c.m.
..	..	..	152.439 sq. c.m.	..	..	152.439 sq. c.m.	..	..	269.028 sq. c.m.	..	..	269.028 sq. c.m.	..	0.235 c.m.
..	..	..	0.24 c.m.	..	..	0.24 c.m.	..	..	0.24 c.m.	..	..	0.235 c.m.	..	0.235 c.m.

* Experiments were made with four different flasks of this glass, with results fairly represented by those given in table. The factor of diminution of resistance for a rise of temperature of 20° C. was about one-fifth.

In our previous paper, the results obtained with eight different samples of lead glass were compared, and it was shown that the electrical conductivity fell off almost quite regularly as the amount of lead oxide increased, and increased with an increase in the amount of soda. The glass which possessed the highest specific resistance (8400×10^{11} ohms) contained 40.5 per cent of lead oxide, 7.5 per cent of potassium oxide, and 2.1 per cent of sodium oxide. Both of the lead glasses dealt with in this paper contained a still higher percentage of lead oxide, and were almost free from soda, and the electrical resistance was so great as not to be measurable. It is, of course, impossible to say how far this result is due to the increase of lead oxide, and how far to the elimination of soda. With the view of definitely settling this point, Messrs. Powell and Sons have kindly undertaken to prepare for us a glass exactly similar to XXI., but having the potash replaced as nearly as possible by the equivalent amount of soda. It should be noticed that the amount of foreign matter (*i.e.*, of ingredients other than silica, lead oxide, and alkali) present in glasses XXI. and XXII. is very small, and is less than one-fourth of the amount present in the purest glass previously tested, which was also, it may be mentioned, the glass possessing the highest resistance.

It is noteworthy that the barium glass XXIII. has a very high resistance, and in this respect behaves more like lead than lime glasses, which have usually a low resistance. It is impossible, however, in view of the somewhat complicated composition of this glass, to say how far the high resistance is due to the presence of the barium, and how far it may be influenced by other ingredients, especially the boric acid, which was not present in any of the glasses previously examined by ourselves or others.

The "Jena" glass XXIV. has a low resistance, as was to be anticipated from its high percentage of soda and complex composition.

The chemical composition of glass XXI. is capable of being expressed with tolerable accuracy by a simple chemical formula, and this is also in accord with previous experience, which pointed to the conclusion that a glass, which approaches in composition to a definite chemical compound, has a high resistance.

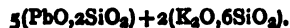
Our knowledge of the chemistry of glasses is still very imperfect, and we cannot say in what way the silica is distributed amongst the bases. We give, therefore, merely the relative number of formula weights of each oxide present, calculated from the analytical numbers, after allowing for the elimination of traces of foreign matter.

Specimen XXI.

After allowing for traces of iron and alumina, the composition of this glass may be expressed by the formula—



or—



	Found.	Calculated.
SiO ₂	50.72	50.53
PbO	42.32	42.33
K ₂ O	6.96	7.13
	100.00	99.99

Specimen XXII.

This glass is essentially a lead-potash glass mixed with a very small quantity of lead-soda glass. Allowing for the small quantity of manganese oxide, arsenious oxide, and other impurities present, we obtain as the simplest expression for the composition of this glass in terms of the formula weights of the oxides present:—



	Found.	Calculated.
SiO ₂	44.67	44.84
PbO	46.78	46.71
K ₂ O	8.03	7.95
Na ₂ O	0.50	0.49
	99.98	99.99

If we eliminate the soda and a corresponding quantity of silica, calculated on the assumption that each formula weight of soda is in combination with three of silica, we obtain as an expression for the composition of the lead-potash glass—



	Found.	Calculated.
SiO ₂	44.07	44.11
PbO	47.72	47.82
K ₂ O	8.19	8.06
	99.98	99.99

Specimen XXIII.

This is a borosilicate of barium and alumina. After allowing for the small quantities of arsenious oxide and manganese oxide which it contains, it has the composition—



	Found.	Calculated.
SiO ₂	33.33	33.35
BaO	48.48	48.20
Al ₂ O ₃	6.06	6.02
B ₂ O ₃	12.12	12.40
	99.99	99.97

Specimen XXIV.

The "Jena" glass is essentially a borosilicate of zinc, soda, and magnesia, containing—



	Found.	Calculated.
SiO ₂	67.87	68.05
ZnO	9.03	9.11
Na ₂ O	10.04	10.46
MgO	5.02	4.50
B ₂ O ₃	8.03	7.87
	99.99	99.99

ON THE OCCURRENCE AND EXTRACTION OF THORITE, MONAZITE, AND ZIRCON.*

By P. TRUCHOT.

(Concluded from p. 135).

As monazite constitutes only a minute portion of the mother-rock it is not, as a rule, practicable to treat it directly. Still some attempts have been made on the sides of the hills, amongst others the Pfeifer mine in Cleveland county, 2 miles north-east of Shelby. The rock is a coarse mica (muscovite and biotite), and the crystals of monazite disseminated throughout it can be plainly seen with a lens. It is very little decomposed, and is still hard. The soil and the subsoil are worked for monazite to a depth of 1.2 to 1.8 metres.

The gravel found is placed in trucks and run to the sluice-boxes. As Mr. Nitze pointed out, when the rock contains gold, monazite constitutes a very valuable by-product.

* *Revue Générale des Sciences*, No. 4, February 28, 1898.

TABLE II.—Average Composition of Monazite and of Monazite Sands.

Formulae.	SAMPLES OF MONAZITE.							MONAZITE SANDS.		
	Mount Ilmen.	Antioquia, New Granada.	Cara- vellas, Brazil.	Burke County, N. Carolina.	Amelia County, Virginia.	Ottawa County, Canada.		Burke County, N. Carolina.	Shelby, North Carolina.	Bellewood, North Carolina.
P_2O_5	27.32	19.13	28.70	29.28	26.12	26.86		18.38	28.16	26.05
Ca_2O_3	31.31	22.88	46.14	31.30	29.89	24.84		32.93 (CeO)		
La_2O_3	31.86	14.69	39.90	30.88	26.66	26.41		7.93	63.80	59.09
Di_2O_3	—	—	—	—	—	—		—	—	—
Y_2O_3	0.52	1.71	—	—	—	4.76		—	—	—
ThO_2	5.55	16.64	—	6.49	14.23	12.60		1.43	2.32	1.19
SiO_2	1.37	9.67	—	1.40	2.85	0.91		6.40	3.20	1.45
Al_2O_3	0.13	2.90	—	—	—	—		1.62	—	0.15
Fe_2O_3	0.26	—	—	—	—	1.07		7.83	traces	0.65
FeO	—	3.56	—	—	—	—		—	—	—
MnO	—	4.89	—	—	—	—		1.20	—	—
CaO	0.55	1.25	—	—	—	1.54		—	—	—
MgO	—	0.40	—	—	—	0.04		—	—	—
ZrO_2	—	—	—	—	—	—		13.98 (+Y ₂ O ₃)	1.52 (+Y ₂ O ₃)	2.68 (+Y ₂ O ₃)
SnO_2	0.95	0.40	—	—	—	—		traces	—	—
PbO	—	—	—	—	—	—		—	—	—
F	—	—	—	—	—	—		—	—	—
Ta_2O_5	—	—	—	—	—	—		0.66	—	6.39
TiO_2	—	—	—	—	—	—		4.67	0.61	1.40
H_2O	0.41	0.71	—	0.20	0.67	0.78		—	—	—

Monazite is thus localised in the west of North Carolina, where the sands richest in thorium are found, in the counties of Burke, Cleveland, McDowell, and the neighbourhood of Bellewood and Carpenter's Knob.

2. *The Deposits of Idaho.*—Last year in the Idaho water-shed, at about 30 miles N.N.E. of Boise city, a large deposit of monazite was discovered, also forming one of the original constituents of the Idaho granite. Certain samples of the sands from the lakes of Idaho City have given, after washing, a monazite sand containing up to 70 per cent of monazite, mixed with zircon, ilmenite, &c.

The alluvial gold washings at Wolf Creek, near Placer-ville, have also yielded large quantities of monazite, mixed with rutile, garnet, &c.

3. *The Canadian Deposits.*—Monazite has been discovered and worked in Canada, at the Villeneuve mine in the county of Ottawa, mixed with mica, garnet, tourmaline, &c.

Deposits in Brazil, Columbia, and the Argentine Republic.

Monazite sands have been found in important deposits in several provinces of Brazil, where they were discovered by M. Gorceix, Director of the School of Mines at Ouro-Preto; in the province of Bahia, at Salabra and at Caravellas; in the province of Minas Geraes, at Diamantina; and in the provinces of Goyaz, Cuyaba, San Paulo, and Rio de Janeiro.

The most considerable deposits in Brazil are found in the province of Bahia, in the form of sand-banks on the sea-shore, particularly at the southern extremity of this province, near the island of Alcobaca.

The continuous destruction of the rocks, by the waves of the sea, has led to a gradual concentration, and we thus find sands of a high average density containing a notable proportion of monazite. The sand is loaded directly on board ship, which enables it to be obtained at a relatively very low price, the actual handling being only the loading. This monazite sand contains an average of 4 or 5 per cent of oxide of thorium, and it almost exclusively supplies the European market.

The other Brazilian deposits have been found in the gold and diamond placer workings in the province of Minas Geraes, Cuyaba, and Goyaz. Monazite is there found in the form of nodular masses, stained and coloured with brilliant yellow particles of orange. Monazite has been met with in the gold placers of Rio-Chico, at Antioquia, in the United States of Columbia; and in the sands of the Buenos Ayres River, in the Argentine Republic.

Table II. shows the average composition of monazite, and of the monazite sands, obtained from various deposits.

Table III. gives the quantity of oxide of thorium contained in a certain number of samples from North Carolina, analysed by Mr. Charles Baskerville. These analyses were carried out on commercial monazite sands containing about 67 per cent of monazite, the remainder being composed of quartz, garnet, zircon, and other minerals.

III.—Zircon.

Zircon, which is always found associated with monazite, and xenotime, &c., is a silicate of zirconium, with the formula ZrO_2SiO_4 .

It crystallises in quadratic prisms, more or less well developed, and shows a large number of modifications. Sometimes they approach the rhomboidal type. Werner described this last variety as *hyacinth*, while the name *zircon* was restricted to the prismatic forms. It is also this latter form which has for many years been called Ceylon Jargon by lapidaries, after the name of the island which then supplied the most beautiful samples of zircons for use as jewels. Similarly to monazite, zircon crystallises in the primitive rocks—granite, syenite, biotite, &c. It possesses a brilliant lustre, is

TABLE III.—Proportion of Thorium in Monazite Sands.

	Oxide of thorium. Per cent.
1. Bennett's Mill, Silver Creek, Barber Co.	0.175
2. North-east Side, Brindle Ridge, Idaho ..	0.225
3. White Bank Gold Mine, Idaho.. ..	2.15
4. Hall's Creek, at Morganton Road, crossing Idaho.. ..	2.25
5. Bailey's Creek, 3 miles S.E. of Glen Alpine Station, Idaho	0.40
6. Linebacher Place, Silver Creek, Idaho ..	6.54
7. MacLewroth Place, Silver Creek, Idaho..	0.125
8. East part of Satterfield Creek, Idaho ..	0.29
9. MacLewroth Branch, MacDowell County	2.48
10. Bracket Town, South Muddy Creek, Idaho	0.26
11. Long Branch, Idaho	1.27
12. Alexander Branch, Idaho	6.30
13. David Peter's farm, Bellewood, Cleveland County	5.19
14. Proctor's farm, near Bellewood, Idaho ..	5.87
15. Wade MacCurd's farm, Carpenter's Knob, Idaho.. ..	6.26
16. Tailings from No. 15	1.75
17. Henrietta, Rutherford County	1.93
18. Fallston, Cleveland County	3.40

transparent or translucent, and of a yellow, grey, brown, red, or hyacinth colour, and sometimes colourless.

Zircon is very widely distributed in Nature, and the most ancient known of its deposits are those of the Island of Ceylon and of Friedrichswarm in Norway, in the province of Christiania. Very beautiful samples are also found in the gemmiferous sands of Espally (Haute-Loire). It is also found at Miask (Ourel), and in the talcous schists of Pfisch (Tyrol). Very important deposits have recently been discovered:—

1. In North Carolina and Colorado.
2. In Texas.
3. In New Zealand.

In North Carolina and Colorado the deposits of zircon are very much mixed up with those of monazite. It is the same with those of Idaho and Texas, of which we have spoken when on the subject of the monazite sands. But all the deposits are small when compared with that which was discovered last year in New Zealand.

This deposit, which covers a superficial area of 105 acres, is situated on the north-east side of Tasmania, half-way between Emin Bay and Circular Head. It is almost entirely composed of zircon, having a density of 4.7 and containing 63 to 64 per cent of oxide of zirconium, with variable quantities of lanthanum, didymium, thorium, niobium, erbium, cerium, and chromium.

It is in a gravel-bed, at about 20 or 30 c.m. below the surface, having a thickness of 20 c.m., that this deposit of zircon is found. It lies on a bed of blue clay of 60 c.m. in thickness, below which there is a bed of sand.

In its exploitation, which is in the hands of a Melbourne Company, large works have been already undertaken; up to the present the zircon is extracted simply by washing and trusting to the differences in density.

Table IV. shows the composition of zircon coming from different deposits.

TABLE IV.—Average Composition of Zircons.

	Formulae.	Ceylon.	Norway.	El Paso, Colorado.	New Zealand.
Silica ..	SiO ₂	33.85	33.61	29.70	33.50
Oxide of zirconium	ZrO ₂	64.25	64.40	60.98	63.80
Protoxide of iron..	FeO	1.08	0.90	9.20	2.07
Magnesia	MgO	—	—	0.30	0.12

We see thus that the deposits of the rare earths tend to increase more and more, and we may easily prophecy that the industry of incandescent gas-lighting, which alone at present actually uses the rare oxides, can never use up the enormous amount of mineral which is now actually obtainable. It is no less true that—thanks to this industry, founded by Dr. Auer von Welsbach—Science has already benefitted by the numerous researches which have enabled us to determine, and partly elucidate, the different characteristics of the rare elements of this group. Never before was it imagined that an industry in rare earths could develop in such a manner and place at the service of Science these tons of rare oxides, such as was shown to be the case by Dr. Otto Witt, on his visit to the Welsbach factory at Gloucester city, New Jersey. This factory treats daily such quantities of monazite sands that M. Witt reports that he actually saw 1000 kilos. of oxide of lanthanum, several tons of salts of cerium, and some hundreds of kilos. of oxide of thorium.

A NEW METHOD OF FRACTIONATION OF THE YTTRIA EARTHS.

By C. URBAIN,

I HAVE studied a large number of the yttria salts from the point of view of their behaviour during fractionation.

The derivatives which have given me the best results by fractional crystallisation are the ethylsulphates. These salts, which form magnificent crystals in the form of hexagonal prisms, have been described by M. Alen and determined by M. Topsøe. They are easily prepared by double decomposition between the yttric sulphates and ethylsulphate of barium.

When fractionated they behave in the following manner:—The first crystals which are formed, though hardly coloured, show a pale orange tint, and are very poor in earths having absorption bands; they also have a lower atomic weight than the mother-liquors.

If the solution contains didymium the didymium accumulates at one end to such a degree that one cannot find it in the other portions of the fractionation, although the ethylsulphate of didymium is very soluble in the absence of the yttria earths. Among the earths of the erbia group found in the first crystals, the element X of Soret is observable with a spectrum decidedly stronger than that of the fresh erbium.

TABLE I.—Yttria Earths from *Æschynite*.

Divisions on the micrometer.	Wave-lengths.	Elements.	First crop of crystals.	Second crop of crystals.
32	654	Erbium.	Faint.	Fairly strong.
34	640	Holmium	Do.	Do.
54—56	582—572	Didymium.	Faint and diffused.	Faint.
67	543	X.	Do.	Do.
69	540	Erbium.	Do.	Do.
72	536	X.	Faint.	Strong.
78	523	Erbium.	Do	Fairly strong, blurred on the right.
79	520	Didymium.	Very faint.	Faint.
100	484	X.	Faint.	Strong.
108	474	Dysprosium.	Very faint.	Faint.
125—130	453—450	Do.	Faint.	Very strong.

This table shows that the fractionation of the yttric ethylsulphates of the monazite sands goes on in a manner very similar to that of *æschynite*. It further shows that fractionation in alcohol is very similar to that in water with regard to didymium, but that the inverse is the case with the yttric earths having absorption bands; these

TABLE II.—Yttria Earths from the Monasite Sands.

Divisions on the micrometer.	Wave-lengths.	Element.	Mother-liquors. Alcoholic solution.	Second crop of crystals.
31	654	Er.	Faint.	Fairly strong.
34	640	X.	Do.	Do.
54—56	582—572	Di.	Very faint.	Faint, blurred.
66	546	Er.	Do.	Faint.
67	543	X.	Do.	Do.
69	540	Er.	Do.	Do.
72	536	X.	Faint.	Strong.
78	523	Er.	Fairly strong.	Fairly strong.
79	520	Di.	Faint.	Faint.
99	484	(X).	Doubtful.	Strong.
101	482	Di.	Do.	Faint.
108	474	Dy.	Do.	Faint.
124—129	453—450	Dy.	Faint.	Strong.

accumulate in the first crops from the alcoholic fractionations.

The spectral study of these portions has been completed by the determination of the atomic weights. By this method of procedure I have confirmed the results found by M. Budischowski and myself (*Comptes Rendus*, 1897) by the fractionation of the acetyl-acetonates, and those obtained by MM. Schutzenberger and O. Boudouard (*Comptes Rendus*, 1897) by the fusion of the nitrates and fractionation of the sulphates. The orange tint of this oxide seems to point to the presence of terbium. Further, the absorption spectrum at this limit, although very faint, shows the presence of at least four substances of high atomic weights.

I should therefore imagine, until the contrary is proved, that this substance is only impure yttrium.

The next crystals deposited in the fractionation of the ethyl-sulphates become more and more pink. The X spectrum of Soret gradually diminishes. The spectrum of dysprosium appears to be more persistent. We next obtain crystals whose solution only gives a very intense spectrum of fresh erbium, and in which the thulium bands seem to be present. The spectrum of the crystals which are finally deposited become gradually fainter, and we finally obtain a mother-liquor which no longer crystallises sensibly and which no longer shows any absorption bands. This earth has a high atomic weight and its oxide is perfectly white.

TABLE III.—Spectrum of the Last Crystals.

Micrometer division.	Wave-lengths.	Elements.	
24	684	Tm (Thulium).	Faint.
31	654	Er.	Faint.
69	540	Er.	Very faint.
78	523	Er.	Strong.
99	485	(X).	Strong.
115—120	469—463	Tm?	Faint.
128	451	Er.	Faint.

In conclusion, we can deduce from the measurements here given that the yttric earths separate in the following order when fractionated as ethylsulphates:—Yttrium, terbium, holmium and dysprosium, erbium, ytterbium.—*Comptes Rendus*, cxxvi., No. 11.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 17th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. A. J. Buller Cooper, J. Murray Crofts, R. S. Morrell, T. Cunningham Porter, F. F. Renwick, and Harold C. Sayer, were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Oscar Julian Steinhart, Ph.D., 4, Palace St. Mansions, S.W.; Samuel A. Tucker, Ph.D., Columbia University, New York; Ernest Witham, The Grammar School, Rotherham.

ANNOUNCEMENT BY THE COUNCIL.

The PRESIDENT announced that notice of the following Resolutions to be proposed at the Annual General Meeting by Messrs. Hartog and Harden had been received:—

Resolution I.

That in order to carry out the suggestion of Mr. Cosens-Hardy, Q.C., communicated by the Council to the Society at the meeting held on February 17th, 1898 (*Proc.*, 1898, xiv., 38), the following steps shall be taken

TABLE IV.—Intermediate Fractions.

Micrometer division.	Wave-length.	Elements.	I.	II.	III.	IV.
24	684	Tm.	Faint, sharp.	—	—	—
27.5	675	Er.	Faint, stronger than 684.	Very faint.	Extremely faint.	—
31	654	Er.	Strong.	Strong.	Fairly strong.	Fairly strong.
34	640	X.	Faint, like 684.	Faint.	Do.	Do.
66	546	Er.	Very faint.	Extremely faint.	Doubtful.	—
69	540	Er.	Fairly strong.	Fairly strong.	Faint.	Very faint.
72	536	X.	Fairly strong, sharp, fainter than 540.	Fairly strong, stronger than 540.	Fairly strong, stronger than 640.	Strong, blurred on the right.
78	523	Er.	Very strong, blurred up to division 80.	Very strong, blurred up to division 80.	Very strong, blurred on the left.	Strong, blurred on the left.
97	489	Er.	Faint, wide, blurred on the left.	Faint, wide, blurred on both sides.	Very faint, wide, blurred.	Extremely faint.
99	485	(X).	Strong, sharp, stronger than 654.	Strong, blurred on the right.	Very strong, blurred.	Strong, nebulous.
108	474	Dy.	Very faint.	Very faint.	Very faint.	Very faint.
117	461	Tm?	Doubtful.	—	—	—
129	451	Dy.	Strong, sharp, blurred on the left.	Very strong, blurred on the left.	Blurred, but the strongest in the spectrum.	Very strong.
136	444	(Di).	Faint, stronger than 489.	Faint, fainter than 489.	Doubtful.	—

to ascertain the wishes of the whole Society with regard to the desirability of obtaining a Supplemental Charter for the purpose of enabling Fellows to record their votes at the Annual Election of the Officers and other Members of the Council by proxy or post.

(1) The following papers shall be printed and distributed to the Fellows of the Society not later than April 14th:—

(a) The Chemical Society, Burlington House, Piccadilly, London, W.

DEAR SIR,—In accordance with the Resolution passed at the General Meeting of the Chemical Society held on March 31st, 1898, we are instructed to request you to be good enough to—

(i.) Answer the question printed below, by writing "Yes" or "No" in the space marked for the purpose.

(ii.) Fold this paper with the blank side outermost, place it in the envelope "A" provided, close the envelope, and write your signature legibly outside.

(iii.) Enclose the envelope "A" in the stamped envelope "B," and return this by post to the Secretaries on or before May 14th next.

We are, faithfully yours,
(Signatures of Secretaries.)

Are you in favour of the proposal that a Supplemental Charter should be applied for to the Privy Council so as to enable Fellows to vote at the Annual Election of the Officers by post or proxy?

(Here write "Yes" or "No").

(b) An envelope marked thus:—

THE CHEMICAL SOCIETY. *Re* CHARTER. A.

Voting Paper.

Please sign legibly here.....

(c) An envelope stamped with a penny stamp, and marked thus:— B.

Re Application for Supplemental Charter.

THE SECRETARIES OF THE CHEMICAL SOCIETY,
BURLINGTON HOUSE, LONDON, W.

(a) At the next meeting held after May 14th the envelopes shall be opened, and the voting papers placed, without being unfolded, in a balloting-box, and they shall be counted by a sufficient number of scrutators, to be appointed by the presiding officer. The signatures on the envelopes shall be at the same time checked against an official list of Fellows of the Society, and shall be preserved for a year.

Resolution II.

If a majority of the votes recorded in accordance with the method prescribed by the foregoing Resolution be in favour of an application being made for a Supplementary Charter for the purpose named, the Council shall be, and are hereby instructed, without any further proceedings, to make the necessary application without delay.

To this notice of Resolutions the following reply had been sent:—

Chemical Society, Burlington House, W., March 17th, 1898.

DEAR SIR,—The Council have carefully considered the proposed Resolutions which you have forwarded to the Secretaries, notifying them that you intend to move them at the Annual General Meeting.

We are, in the first place, to point out that the effect of passing such Resolutions would be precisely the same as passing a Bye-law to enable Fellows to vote by proxy or through the post on this question, and that Mr. Cozens-Hardy, Q.C., has already advised that any such Bye-law would be repugnant to the Charter.

We are directed to say that this of itself would be sufficient to make the proposed Resolutions out of order, but there are other grounds on which the Council are advised that such Resolutions cannot be submitted to a General Meeting.

The Council are advised that in view of the opinions of

Mr. Cozens-Hardy, such Resolutions would not be in order, because they propose to deal with a question which is outside the scope of matters which can be decided at a General Meeting of the Society.

They are also advised that even if such Resolutions were passed they would have no power to act upon them, because in considering whether the Fellows really desire the change proposed, regard must not be had only to those who are present in person at a meeting (and still less to a bare majority of Fellows voting, as you propose), but to the wishes of the whole body of the Fellows. You will remember that Mr. Cozens-Hardy thinks that an application for a Supplemental Charter would probably not be listened to unless it represented the practically unanimous view of the Fellows, and that any active opposition by even a small minority would probably be fatal.

The Council are further advised that it would be *ultra vires* to expend any part of the funds of the Society in giving effect to the proposed Resolutions or in applying for a Supplemental Charter.

In these circumstances, we are directed to inform you that it would be the President's duty to refuse to put such Resolutions to the meeting.

We are also to point out that it would be open to any Fellows of the Society, at their own expense, to send out the circulars referred to in the Resolutions, and that if it should be found that the Fellows were practically unanimous in desiring that application should be made for a Supplemental Charter, this could be applied for by them at the like expense, and that in the event of there being no opposition Her Majesty might be pleased to direct that such expenses should eventually be paid out of the funds of the Society.

We are, dear Sirs,
Your obedient Servants,
JOHN M. THOMSON, } *Honorary*
WYNDHAM R. DUNSTAN, } *Secretaries.*

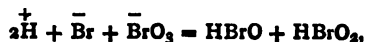
Arthur Harden, Esq.

P. J. Hartog, Esq.

Of the following papers those marked * were read:—

*34. "The Reduction of Bromic Acid and the Law of Mass Action." By WINIFRED JUDSON, B.Sc., and J. WALLACE WALKER, M.A., Ph.D.

The authors have investigated the velocity of the reduction of bromic acid by hydrobromic acid, and find that, in the presence of a large quantity of sulphuric acid, the reaction is bimolecular, whereas in the absence of sulphuric acid it is tetramolecular. The results are in agreement with the conclusions drawn by Noyes (*Zeit. Phys. Chem.*, 1896, xix., 599) from the experiments of himself and others on the reduction of bromic acid by hydriodic acid. The explanation put forward is that the stage of the reaction, which requires a measurable time for its completion, is expressed by the equation—



and that the bromous and hypobromous acids are instantly decomposed by the hypobromic acid present. A further result obtained is that the reduction of bromic acid by hydriodic acid takes place 58.5 times faster than by hydrobromic acid, and that, therefore, although the latter is produced from bromic by hydriodic acid, its presence in no way vitiates the conclusions drawn by Noyes from that reaction.

*35. "The Action of Ferric Chloride on the Ethereal Salts of Ketone Acids." By R. S. MORRELL, M.A., Ph.D., and J. M. CROFTS, B.A., B.Sc.

When anhydrous ferric chloride dissolved in absolute ether is added to an ethereal solution of ethyl ketophenylparaconate, a red oil separates. This, when washed with absolute ether, becomes solid, and is, most probably, represented by the formula $\text{FeCl}_2(\text{C}_{13}\text{H}_{11}\text{O}_3)_2$. Water decomposes the compound with formation of

the basic ferric salt of ethylic ketophenylparaconate, $\text{Fe}(\text{OH})(\text{C}_{13}\text{H}_{17}\text{O}_5)_2$, and ferric chloride.

The ethylic salt of the lactone of oxalctic acid yields with ferric chloride in absolute ether solution a red oil, which does not solidify, and is most probably an addition product, represented by the formula $\text{FeCl}_3 \cdot \text{C}_{14}\text{H}_{18}\text{O}_9$. It loses hydrochloric acid slowly in a vacuum. On treatment with water, it yields the ferric salt of the lactone of ethylic oxalctrate, $\text{Fe}(\text{C}_{14}\text{H}_{17}\text{O}_9)_3$, and ferric chloride.

In the case of ethylic acetoacetate and ethylic benzal-di-acetoacetate, purple oils are obtained by the action of anhydrous ferric chloride. The purple substances have not as yet been prepared in a sufficiently pure state to justify the authors in assigning formulæ to them.

*36. "Note on the Volatility of Sulphur." By T. C. PORTER, M.A., &c.

During the analysis of some pumice rock from Tenerife, the author was led to the conclusion that sulphur is volatile at 100° . Several glass tubes were carefully cleaned, into which specimens of sulphur of varying physical condition and purity were placed. Some of the tubes were exposed to the air, others were exhausted. After being exposed to a temperature of 100° for some minutes, it was observed that, as was to be expected, the better the vacuum the more rapid is the sublimation of the sulphur. The sublimate consists at first of very pale yellow drops, which possess little viscosity, and remain unchanged in some cases even at 10° for days. Rhombic octahedra and prismatic sulphur are occasionally formed together; the former crystals sometimes grow at the expense of the drops. Sulphur does not yield a perceptible sublimate at ordinary temperatures, in a "good vacuum," even after a year. The two forms S_8 and S_6 therefore have their transition point between 90° and 100° .

DISCUSSION.

Professor McLEOD said that about 1870 Mr. Douglas Herman had experimented on the volatility of various substances in vacuum tubes, although he believed the results have never been published. Mr. Herman found that sulphur could be volatilised in a vacuum when heated to the temperature of boiling water, and that the vapour condensed in drops on the cool parts of the tube, and remained liquid for many days. Octahedral crystals were formed at the expense of the drops in the neighbourhood of the crystals, the drops gradually evaporating whilst the crystals increased in size; prismatic crystals were very rarely seen. When phosphorus is heated *in vacuo* by the warmth of the hand, its vapour also is deposited in drops, although, as in the case of sulphur, the vapour had not been heated to the melting-point of the solid. Iodine at once forms crystals under similar conditions, no liquid being deposited.

The PRESIDENT said that, in a lecture on liquid atmospheric air, in 1893 (*Proc. Roy. Inst.*, xiv., 7), he had described the use of sulphur in the construction of vacuum vessels for the storage of liquid air, and had shown that in such vacua cooling with liquid air or oxygen was sufficient to produce a visible distillation of sulphur at ordinary temperatures. The vapour tension of sulphur at 100° amounts to 0.06 m.m., so that the tension is comparatively high in Mr. Porter's experiments, considering that mercury distils rapidly under a pressure of one-millionth of an atmosphere, as shown by the application of liquid air. He also remarked that phosphorus distils practically instantaneously.

*37. "Cannabinol." By T. B. WOOD, M.A.; W. T. N. SPIVEY, M.A.; and T. H. EASTERFIELD, M.A., Ph.D.

The authors have continued their examination of cannabinol, the toxic resident constituent of Indian hemp (*Trans.*, 1896, lxi., 539). The substance boils with slight decomposition at about 400° , its absorption spectrum shows no characteristic bands, its vapour-density at the temperature of boiling sulphur corresponds with the formula $\text{C}_{18}\text{H}_{24}\text{O}_2$, already assigned to the compound.

An account is given of the reaction of cannabinol with acetic anhydride, benzoyl chloride, and phosphoric anhydride; the results indicate that one hydroxyl group is present. In the case of acetic anhydride or acetyl chloride, however, a crystalline compound melting at 75° is one of the products of the reaction. The authors assign the formula $\text{C}_{17}\text{H}_{23}\text{O}_2$ to this compound. The same compound has recently been described by Dunstan and Henry (*Proc.*, 1898, xiv., 44, Feb. 17), who ascribe the formula $\text{C}_{18}\text{H}_{23}\text{OAc}$ to it. Fuming hydriodic acid gives no methyl or ethyl iodide when boiled with cannabinol. Reduction with hydriodic acid in sealed tubes produces a hydrocarbon, $\text{C}_{10}\text{H}_{16}$.

By long-continued boiling, with or without dehydrating agents, a hydrocarbon, $\text{C}_{10}\text{H}_{16}$, is formed.

Oxidation with aqueous chromic acid, alkaline, or acid permanganate, or dilute nitric acid, is accompanied by the production of a caproic acid, lower fatty acids being probably produced at the same time. The action of fuming nitric acid upon cannabinol dissolved in cold glacial acetic acid removes one carbon atom as carbonic anhydride, and produces a red, amorphous substance, which gives numbers on analysis agreeing with the formula $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_6$. This substance, when boiled with nitric acid, yields a light red substance, $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_8$, which upon further oxidation yields, amongst other substances, a yellow acid crystalline compound, $\text{C}_{13}\text{H}_{15}\text{N}_2\text{O}_5$, which forms sparingly soluble crystalline sodium, ammonium, and silver salts, and is probably a dinitrophenol, and a compound, $\text{C}_{11}\text{H}_{11}\text{NO}_4$, the properties of which agree closely with those of the "oxycannabin" of Bolas and Francis (*CHEM. NEWS*, 1871, xxiv., 77). This compound has the properties of a nitro-lactone, as has been already shown by Dunstan and Henry (*Proc.*, loc. cit.). Corresponding crystalline potassium and silver salts have been prepared and analysed. The name cannabinic acid is proposed for the unnitrated parent oxy-acid.

Amido-cannabinolactone, $\text{C}_{11}\text{H}_{11}\text{O}_2\text{NH}_2$, is obtained in colourless crystals melting at 119° , when the nitro-lactone is reduced either by hydriodic acid or by tin and hydrochloric acid. The base is readily re-crystallised from hot water; its salts cannot be re-crystallised from water without decomposition; the hydriodide and platinochloride have been analysed.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, March 25th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

Mr. A. A. CAMPBELL SWINTON read a paper and showed experiments upon "The Circulation of Gaseous Matter in a Crookes Tube."

The stream-lines within a Crookes tube are investigated by observing the direction and speed of rotation of a mica radiometer-mill, mounted on a sliding rod, so that it can be moved along a line at right-angles to the line joining the electrodes. The axis of the mill is at right-angles to both these lines. If the mill is adjusted to a position between the flat-plate and the cup electrodes, with its axis just sufficiently low to prevent equal and opposite simultaneous action on the top and bottom vanes, it rotates always in the direction indicating a stream from cathode to anode; the speed is greater when the flat plate is the cathode. If, however, the mill is now moved below this line, a point is reached at which rotation ceases, and below this neutral point the rotation is suddenly reversed. Reversal is only to be observed with high degrees of exhaustion; the rotation is never so rapid as in the first position. The mill rotates and the reversal may be observed whether cup or plate is made cathode, and the direction of rotation below the neutral point is always opposite to that in the position above it. A small

Wimshurst machine is as effective as an induction coil in producing these effects.

The experiments are intended to establish the existence, at high degrees of exhaustion, of a true anode-stream, i.e., a stream that travels from anode to cathode just in the same manner as the cathode stream flows from cathode to anode. This anode-stream is charged positively; it is exterior to the cathode-stream; its velocity is less than that of the cathode-stream, but its velocity increases as the vacuum is improved. It seems probable that, in high vacua, some portion of the positive electricity passing through the tube is carried by the positively charged atoms or particles that constitute the anode-stream. At lower degrees of exhaustion, the discharge passes through the tube chiefly by interchange of charges from molecule to molecule—a Grothūs chain. At very high vacua, however, when the mean free path is considerable, there may be to some extent a regular and complete circulation of positive and negative atoms, some of which pass from anode to cathode and *vice versa*, and deliver up their charges, not by interchange, but by direct convection, to the electrodes of opposite sign.

Prof. Boys said he did not feel altogether convinced by the experiments that the rotation of the mill was due to simple mechanical motion of the particles of matter between the electrodes. The weight of air left in the tube at such high degrees of exhaustion was extremely small; it was difficult to realise that its impact could produce the sudden mechanical effect observed at the moment of the reversal of the rotation of the mill.

Mr. WIMSHURST thought it important to keep in mind the existence of mercury vapour in the tube. He also referred to some experiments in which a bar of metal was used to explore a focus tube, by observations of the changes of luminosity produced in different positions.

Dr. CHREE said that if the rotations of the mill could be shown to indicate a velocity of the particles, of the same order as that observed in Crookes's experiments, it was safe to assume the existence of a similar cause. This might be important in deciding as to the general truth of the bombardment theory of Crookes. He asked whether the rotation had been investigated within the dark space around the cathode.

Mr. APPLEYARD suggested that in tracing the cause of the rotation, it would lead to simpler results if the vanes of the mill were made of some light conducting substance. Mica introduced difficulties, owing to its retention of the charges.

Prof. Boys pointed out that this could be done by gilding the mica.

Mr. CAMPBELL SWINTON, in reply, said that the objection raised by Prof. Boys to the mechanical theory of the rotation would apply equally to the whole theory of electro-radiometry, including the case of the mill used originally by Crookes in the direct path of the cathode stream. But it must be remembered that, although the mass of matter present within the tube was very small, its velocity was proportionally great; it was of the order of 9000 kilometres per second. Hence, the contained matter might be conceived as capable of producing the observed acceleration, and Crookes's bombardment theory might safely be adopted as a good working hypothesis. In the tubes used for these experiments, the exhaustion was carried so high that the negative dark space appeared to fill the whole tube. He had, so far, only tried mica for the vanes, but he thought it would be important to observe the results with a substance that did not retain the charges.

Mr. A. STANSFIELD then read a paper on "*Thermo-electric Pyrometers*."

In obtaining photographic records of the readings of thermo-electric pyrometers, the range of measurement is limited by the size of the photographic plate. For long ranges of temperature, the sensitiveness of the galvanometer must therefore be small.

When it is desired to examine the temperature changes

in detail—as, for instance, at the melting-points and freezing-points of metals—it is necessary to employ some device for giving a more open scale for the short temperature ranges that include those particular points. For this purpose two galvanometers are arranged in parallel, and so that they have their deflections recorded on the same photographic plate. The less sensitive galvanometer covers the entire range of temperature throughout an observation; the other is brought into use for magnifying special portions of the range. In this latter case part of the electromotive force of the thermocouple is compensated by an opposing electromotive force, applied at two points of the circuit, from a battery of Clark cells in series with a high resistance.

The recording apparatus consists of a photographic plate mounted on a float that rises steadily when water is admitted into a cylinder. The source of light is a glow-lamp, enclosed in a wooden box. A brass tube with a rectangular diaphragm at the end nearest the lamp cuts off all light, except that from a selected piece of vertical filament. Light from this filament is reflected by the plane galvanometer-mirror, and is focussed upon the photographic plate by a lens in front of the galvanometer: this method was suggested by Prof. Boys. The "cold" junctions of the thermo-couple are both inserted into a hypsometer. Very serious discrepancies exist between the indications of couples having nominally the same composition; they are too great to be attributed to accidental differences in the constitution of the alloys. Although with platinum alloys, coupled with platinum, 10 per cent of iridium gives a more powerful couple than 10 per cent of pure rhodium; the partial substitution of iridium for rhodium very considerably lowers its thermo-electric power. This result suggests that the change in the thermo-electric power of a metal depends upon the extent to which it is saturated with the alloying metal; thus 10 per cent either of rhodium or iridium would more completely saturate the platinum than would 10 per cent of a mixture of the two metals. The author discusses a series of curves derived from his experiments. He concludes that, thermo-electrically, there may be two classes of metals:—(1). The ordinary metals, for which the curve representing the first differential of electromotive force with respect to temperature is a straight line; and (2). The platinum metals, together with a few such as nickel and cobalt, for which the curve of this differential multiplied by the absolute temperature is a straight line.

Dr. CHREE discussed the curves, and asked how far stirring affected the results; he was inclined to think that stirring was a mistake.

Mr. A. CAMPBELL enquired whether the galvanometer kept its zero sufficiently well throughout the tests.

Mr. STANSFIELD, in reply, said he had also come to the conclusion that stirring was a mistake; it was a mistake to use a large quantity of metal. The pyrometers were sensitive to about a tenth of a centigrade degree. He had experienced great difficulty with regard to the zero of the galvanometer.

The PRESIDENT proposed votes of thanks to the authors, and the meeting adjourned until April 22nd.

CHEMICAL NOTICES FROM FOREIGN SOURCES

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Berichte der Deutschen Chemischen Gesellschaft.
1898, No. 1.

The New Development of the Lighting Effect of Flames.—H. Bunte.—Contains a history of the gas industry, the introduction of glow lamps, and the discovery of acetylene from calcium carbide.

The Products of the Explosive Decomposition of Nitro-Compounds.—Christian Göttig.—The author points out that the numbers given for the composition of the gases formed by the explosion of gun-cotton by different investigators were very variable. He therefore makes a careful investigation of the solid and gaseous products formed by the explosion of gun-cotton having the composition, barium nitrate 9.83 per cent, nitro-toluol 22.22 per cent, and nitro-cellulose 67.96 per cent. The solid products consist of carbon 9.51 per cent, barium carbonate 64.44 per cent, and a residue insoluble in acid 26.05 per cent. The gaseous products consist of oxides of nitrogen 10.75 per cent, carbon dioxide 27.48 per cent, carbon monoxide 36.02 per cent, methane 9.01 per cent, hydrogen 1.94 per cent, and nitrogen 14.8 per cent.

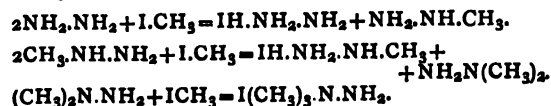
The Action of Benzhydrazide on Glucose.—Georg Pinkus.—The author finds that in alkaline solution glucose is decomposed, in presence of benzylhydrazide, into glyoxal and methylglyoxal, which are removed from further action by reason of the insolubility of the osazone formed. He suggests that the methylglyoxal is an intermediate product in the long-known formation of lactic acid from sugar in alkaline solution.

Constituents of Beech-Tar.—C. Harries.—Alterberg has isolated from the volatile parts of the tar oil of *Pinus sylvestris* a body for which he found the formula $C_9H_{10}O$. He recognised this as methylfuran, and gave to it the name Sylvan. The author has now found that this body can also be obtained as an ingredient of beech-tar creosote (the part boiling between 60° and 70°). To get rid of aldehyd and ketone from this substance it is treated with sodium bisulphite, the remainder shaken up with NaOH solution, and then dried with K_2CO_3 ; it is then fractionated. The sylvan boils at 65° under 759 m.m. pressure, and has sp. gr. 0.827 at 18° . It is a light, mobile, colourless liquid, of ethereal smell, which, after standing for twenty-four hours, becomes bright yellow. This colour can be removed by treating it with a very small quantity of alcoholic HCl.

Some Syntheses with Chlorfumaric Ester.—Walter Beckh.—The condensation of ethenyltricarboxylic ester and chlorfumaric ester by means of sodium methylate gave, as chief product, a butylene pentacarboxylic ester. The free acid was only obtained in a very impure condition. By further action of sodium methylate, a molecule of alcohol split off and a substance was obtained to which the author assigns the name tetrylone-tetracarboxylic ester. Amylene pentacarboxylic ester was prepared in a similar way by condensation of chlorfumaric ester with sodium carboxyglutaric ester, and when treated with sodium ethylate it also lost a molecule of alcohol, yielding a reduced benzene compound.

Nitrogen-free Decomposition Products of Morphine.—E. Vongerichten.—The author finds that phenanthrene derivatives can be obtained from morphine derivatives, not only on distillation with zinc dust, but also with chromic and acetic acids. He obtains a phenanthrene quinone derivative. Acetylmethyl morphol from methylmorphmethane gives a very good yield of acetylmethylmorphol quinone, which shows that morphine bears a very close relation to phenanthrene.

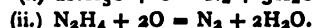
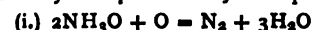
Methylating of Hydrazin Hydrates.—C. Harries and T. Haga.—(a) Action of Methyl Iodide on Hydrazin Hydrate in the presence of KOH.—If hydrazin hydrate is shaken up with methyl iodide, in the cold, hydriodide of hydrazin is formed, which can be decomposed by KOH, and again treated with methyl iodide. The reaction may be represented by the following equations:—



Trimethylazonium iodide is somewhat soluble in alcohol

and amyl alcohol on boiling; it crystallises in the same way as NH_4Cl , and decomposes before melting at about 235° . (b) Action of Methyl Iodide on Hydrazin Hydrate without KOH.—With excess of hydrazin hydrate, methyl hydrazin and dimethyl hydrazin are formed, but no azonium salt or a very little. When, on the contrary, excess of methyl iodide is used, besides hydrazin hydriodide, trimethylazonium iodide is obtained.

A Method of Gravimetrically and Gasometrically estimating Hydroxylamine and Hydrazine.—K. A. Hofmann and F. Kuspert.—The authors estimate hydrazin as well as hydroxylamine, through oxidation with dilute vanadic solution containing H_2SO_4 . The evolved N is collected, and the partially reduced solution is titrated back to vanadic sulphate, by means of permanganate. The reaction may be represented by the equations:—



On warming the solution to about 60° the N comes off quantitatively, and can be measured. The contents of the flask are titrated with permanganate solution, and this result can be used to check the volumetric measurement of the nitrogen.

Nitration of Carbohydrates.—W. Will and F. Lersze.—The authors have made experiments on the nitration of the whole class of sugars, and have succeeded in obtaining pure and in many cases crystalline products. From glucose and xylose they were unable to obtain crystalline products. Generally the best results were obtained when HNO_3 at 0° (sp. gr. 1.52) was added to the finely-powdered substance until it was dissolved, and cold concentrated H_2SO_4 (sp. gr. 1.84) dropped slowly on to the solution. The following products were obtained:—(1) Tetranitrates of the pentoses; (2) of the hexoses, crystalline pentanitrates of glucose, galactose, and mannose, and a trinitrate of laevulose; (3) hexanitrates of heptose. Among the disaccharides, cane-sugar, milk-sugar, and maltose are found to give octonitrates as higher products of nitration.

Chemiker Zeitung.
1898, No. 11.

Formation of Perchlorates of the Alkalies and Alkaline Earths by Electrolysis.—F. Winteler.—By using platinum or peroxide electrodes, aqueous solutions of the chlorides of the alkalies and alkaline earths can be electrolysed, hypochlorites and chlorates being formed. Under certain circumstances the perchlorates can be formed also. References are found in the literature on the subject to the turning of $KClO_3$ into $KClO_4$ between platinum electrodes; but it is remarkable that none of the patents for the preparation of chlorate by electrolysis take this into consideration, and in the new books it is passed over completely. When the electrolysis has proceeded so far that only chlorate is found in the solution, this chlorate will oxidise further between platinum or peroxide electrodes under the following conditions:—(1) When there is an acid solution round the anode. (2) When the anode is at a low temperature. (3) When there is a current density of 4–12 amperes. (4) When the concentration of the electrolyte is as great as possible. Suppose, for example, at ordinary temperature, a saturated neutral solution of $KClO_3$ is electrolysed without using a diaphragm, the electrodes being kept at 10° , 75 per cent of the oxygen which ought to separate out is used to convert the chlorate to perchlorate. A saturated $NaClO_3$ solution (1:1), under exactly similar circumstances, is transformed into perchlorate with a yield of 95 per cent. When nearly the whole of the chlorate is transformed, the yield begins to decline very rapidly. The direct cooling of the electrode is of great importance. The apparatus in which the electrolysis takes place must be so arranged that the electrode comes in contact with

the electrolyte on one side only, otherwise the current density cannot be indicated accurately, for only the side turned to the cathode comes into consideration. The cathode density is immaterial, and depends only on the voltage. Other things being equal, the yield is the greatest when the anode current density is 8–12 ampères. Greater or less density diminishes it. Even a chlorate solution, to which 4 per cent of alkali has been added, changes into the required perchlorate. The stirring up of the anode and cathode solutions is not done mechanically, but by means of the evolved gas. An intense smell of ozone is characteristic of the formation of perchlorate. Ozone is evolved in such quantities from a concentrated chlorate and perchlorate solution that rubber tubing and connections will be destroyed in a few minutes, and it would be worth while to use this electro-chemical method for preparing ozone. If carbon is used for the electrodes it is very quickly destroyed, no formation of perchlorate taking place. The use of any other metal than platinum or iridium is out of the question, since the anode takes away ClO_2 and forms a soluble salt with it. An interesting observation, which has been lost sight of recently, is that of A. Riche, by which dilute HCl is turned into HClO_4 by electrolysis between platinum electrodes. According to my experiments, the conditions favourable for this are identical with those of the formation of the perchlorate. The perchloric acid will be more concentrated the lower is the temperature of the electrolyte—the best of all being a temperature at which no gaseous chlorine can escape.

MISCELLANEOUS.

The Chemical Laboratory at Wiesbaden.—The hundredth term at the Chemical Laboratory Fresenius will shortly begin. The Laboratory was established on the first day of May, 1848, and the celebration of the fifty years of its existence will shortly take place. Its founder, Geh. Hofrath Professor R. Fresenius, died on June 11th last, and since his death the Laboratory has been carried on by his sons, Professor Dr. Heinrich Fresenius and Dr. Wilhelm Fresenius, and his son-in-law, Dr. Ernst Hintz, in co-partnership; they will continue the institution in the same manner as before. The list of the enrolled students of the Winter Session 1897–98, just published, shows an equal attendance at the Laboratory as in former years. There were fifty-three students on the books during the Winter Term 1897–98. Of these, forty were from Germany, four from England, two from Austria, two from Luxemburg, one from France, one from Spain, one from Sweden, one from Russia, and one from Brazil. Of the students, forty-four were working exclusively in the chemical laboratory, seven exclusively in the bacteriological department, and one both in the chemical and bacteriological branches of the Institute. There were employed three assistant teachers in the instructing laboratories, and twenty-five assistants in the different departments of the Laboratory (Versuchsstationen). Besides the Directors (Professor Dr. H. Fresenius, Dr. W. Fresenius, and Dr. E. Hintz), Messrs. G. Frank, M.D., W. Lenz, Ph.D., L. Grünhut, Ph.D., and architect J. Brahm, belong to the faculty of the College. The next Summer Term begins on April 25th. During the last term, besides the scientific researches, a great number of analyses were undertaken in the different departments of the Laboratory on behalf of manufacture, trade, mining, agriculture, and hygiene.

The Titration of Stannous Salts with Iodine.—S. W. Young.—The author states that the reaction between stannous chloride and iodine, by which the former is oxidised, takes place smoothly in acid solution, and may be utilised for the determination of tin. The iodine

solution is standardised against potassium bichromate with the intervention of a stannous chloride solution of known value. The statements are made that "thio-sulphate cannot be satisfactorily used in acid solution to titrate against iodine," and that "the standard of iodine by thiosulphate is likely to be a trifle high;" but no authorities are quoted nor experimental data offered in support of these assertions.—*Journ. Amer. Chem. Soc.*, xix., 809.

The Analysis of Raw Sulphide of Sodium.—F. Jean.—The crystals of raw sulphide of sodium are crushed, and 10 grms. dissolved in water and diluted to 1 litre. To 10 c.c. of this solution starch is added; it is then titrated with decinormal iodine. To another 10 c.c. of the original solution a quantity of sulphate of ammonium (at 6.7 grms. per litre) is added, in quantity equal to the number of c.c. of the solution of iodine used in the first case; then add about 30 c.c. of water and distil over the ammonia set free by the monosulphide of sodium. Then boil to drive off the sulphuretted hydrogen, and titrate the excess of acid: 1 c.c. of decinormal acid saturated with ammonia corresponds to 0.0039 gm. of monosulphide of sodium. The liquid remaining in the retort is titrated with a N/10 solution of iodine: 1 c.c. of the iodine corresponds to 0.0079 gm. of hyposulphite of soda.—*Journ. de Pharm. et de Chim.*, vii., No. 4.

The Solubility of Theobromine in Aqueous Solutions of Salts of an Alkaline Reaction.—M. Brissemoret.—At a temperature of $+15^\circ$ a solution of 14.80 grms. of trisodic phosphate in 80 c.c. of water will dissolve 3.50 grms. of theobromine. One would at first be inclined to think that a combination took place between the trisodic phosphate and the theobromine, as these weights agree with their molecular weights. As a matter of fact, this phenomenon is simply the solution of the theobromine in the alkali set at liberty by the dissociation that the phosphate of soda undergoes on contact with water.—*Journ. de Pharm. et de Chim.*, vii., No. 4.

MEETINGS FOR THE WEEK.

MONDAY, 4th.—Society of Chemical Industry, 8. "The Bacterial Treatment of Sewage containing Manufacturing Refuse," by W. J. Dibdin, F.I.C., F.C.S.
TUESDAY, 5th.—Society of Arts, 8. "The British Empire—its Resources and its Future," by John Lowies, M.P.

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W. FRESSENIUS, Ph.D.
E. HINTZ, Ph.D.

LECTURES.

Experimental Chemistry (Inorganic) Prof. H. FRESSENIUS, Ph.D.
Experimental Physics W. FRESSENIUS, Ph.D.
Stoichiometry L. GRÜNHUT, Ph.D.
Organic Chemistry W. LENZ, Ph.D.
Chemical Technology Prof. H. FRESSENIUS, Ph.D.
Microscopy, with exercises in Microscopic work W. FRESSENIUS, Ph.D., and E. HINTZ, Ph.D.
Chemistry and Analysis of Foods Dr. med. G. FRANK.
Hygiene J. BRAHM.
Practical exercises in Bacteriology
Technical Drawing, with exercises

The next Session commences on the 25th of April. The Regulations of the Laboratory and the Syllabus of Lectures will be forwarded gratis on application to C. W. KREDEL's Verlag, at Wiesbaden, or to one of the Directors.

CHEMICAL AND ALLIED TRADES

EXHIBITION AND MARKET, 1898.

An Exhibition of the Chemical and Allied Trades will be held at the Agricultural Hall, London, N., from the 13th to the 17th June, 1898, at the same time as the 4th Annual Chemists' Exhibition, and under the same management.

The Chemists' Exhibition is one of the most successful of the trade exhibitions held yearly in London, and is visited annually by a very extensive trade assembly, which includes large numbers of Manufacturing Pharmacists, Wholesale Druggists, Analytical Chemists, Makers of Proprietary Articles, Exporters of Drugs and Chemicals, and numerous Foreign and Colonial buyers.

By taking advantage of this opportunity, the presence of a considerable number of extensive purchasers and consumers of products connected with the Chemical and Allied Trades is assured, and as the Exhibition is being extensively advertised to all interested in these trades throughout the United Kingdom and the Continent of Europe, a very large attendance of those with whom supplying houses desire to do business can be confidently anticipated. Exhibitors will thus have an opportunity to submit their specialities to the inspection of very large numbers of trade buyers, under the conditions most favourable to business, and in circumstances of advantage such as are not afforded to them in this country on any other occasion.

Plans and further particulars can be obtained from Mr. J. F. CANTWELL, Managing Director of the Chemists' Exhibition, 44, Bishopsgate Street Without, London, E.C. Personal enquiries can be made between 11 and 1 o'clock any day except Saturday.

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£1 9 0

No. 85. Fig. 7. STUDENT BALANCE, in french polished mahogany glass case, with counterpoised front sliding frame, steel knife edges, and needle pointer, with nickel pans, to carry 30 grammes in each pan and turn to half a milligramme, but with flat pans £2 9 0



FIG. 11. No. 91.

No. 91. Fig. 11. SHORT BEAM BALANCE for a charge up to 100 grammes in each pan, in french polished glass case, front sliding frame counterpoised, beam divided on both sides in 1-5th part of milligrammes, with new improved arrangement for arrest of pans. Provided with wood stand for taking specific gravity, agate bearings, and sensible to 1-10th part of a milligramme, pans 6 centimetres in diameter, width of bows 9 centimetres. Pans and bows nickel, provided with two rider apparatus, with agate bearings £9 0 0

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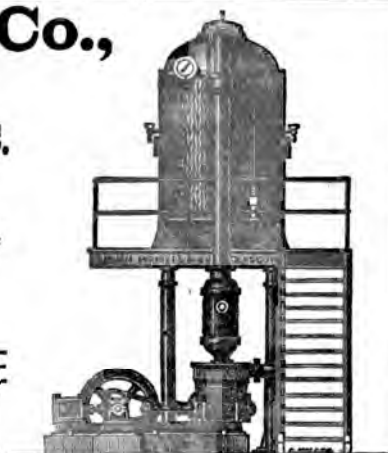
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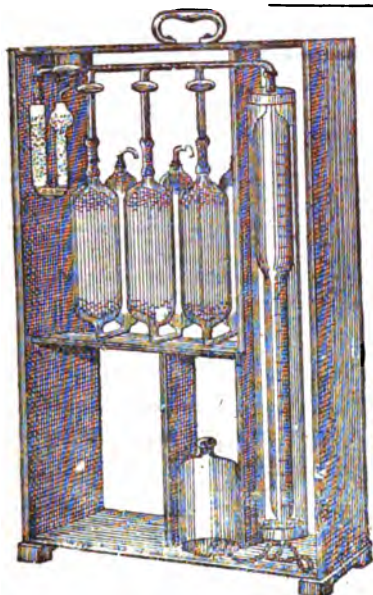
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CONTENTS.

ARTICLES:—	PAGE
On the Composition of the Atlantic Ocean, by C. J. S. Makin, F.I.C., F.C.S.	155
Note on the Volumetric Estimation of Chromium, by Rudolf L. Leffer, F.C.S.	156
The Cyclical Law of the Elements, by Thomas Bayley, Assoc.R.C.Sc.	157
PROCEEDINGS OF SOCIETIES:—	
CHEMICAL SOCIETY.—Contributions to the Chemistry of Thorium—On the Atomic Weight of Thorium—On the Compound Nature of Cerium—On Praseodymium and Neodymium—Action of Ammonia and Substituted Ammonias on Acetylurethane—Formation of Oxytriazoles from Semicarbazides—Formation of $\alpha\alpha'$ -Dihydroxypyridine—Position-isomerism and Optical Activity; the Comparative Rotatory Powers of Diethyl Mono-benzoyl and Mono-tolyl Tartrates—The Action of Di-isocyanates upon Amido-compounds—The Action of Alkyl Iodides on Silver Malate and on Silver LaGate—On the Optical Rotations of Methyl and Ethyl Tartrates	160
NOTICES OF BOOKS.—Agriculture in some of its Relations with Chemistry.	163
CHEMICAL NOTICES FROM FOREIGN SOURCES	164
MISCELLANEOUS	166

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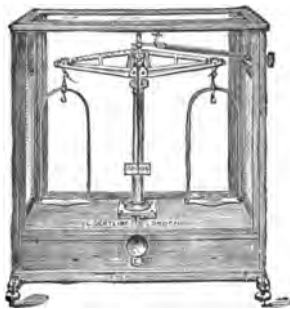
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ON THE COMPOSITION OF THE ATLANTIC OCEAN.

By C. J. S. MAKIN, F.I.C., F.C.S.

DURING a recent voyage from the Cape samples were drawn daily about noon in the neighbourhood of the ship's position. These individual samples and their solid contents per 1000 grms. are tabulated below. The whole series (excluding No. 11) were averaged, and the resulting mixture subjected to an exhaustive examination. The analyses were, as far as the writer found practicable, conducted on the same lines as those of the *Challenger* by Professor Dittmar.* Determinations of ammonia and organic matter have also been made: owing to the samples of the *Challenger* waters running short, these estimations were not made on that occasion.

The specific gravity of the average sample was found to be 1.0275 at 15.5°.

Chlorine and Total Solids.

The halogen contents were determined by ordinary volumetric means. The volume (25 c.c.) for determination was delivered from a calibrated burette. The same volume and burette were employed for all the other determinations, excepting bromine and carbonic acid. Gravimetric means were also tried, but abandoned as probably falling short of the truth.

By volumetric means were obtained—

20.729
20.722
20.721

Mean, 20.724 halogens per litre.

By gravimetric means were obtained—

20.713
20.719

Mean, 20.716

20.7240 was finally adopted. Subsequent bromine determinations gave 67.10 m.grms. Hence, deducting the latter figure, 20.6569 grms. chlorine per litre are obtained.

The individual samples were titrated under the same conditions. Professor Dittmar found it quite impossible to arrive at a true estimate of the total solids by the ordinary method of evaporation and drying. Ultimately, the factor 1.8058 was adopted, as it was proved that, by multiplying the total chlorine by it, a figure was obtained giving the total solids with an error of ± 0.002 per kilo. This factor has been adopted in the tabulated results and the figures thereby obtained reduced to the proportion per 1000 grms.

A study of the relation of the chlorine to the specific gravity resulted in a series of tables from which the average chlorine may be deduced from any given gravity. With the found gravity (1.0275), a total of 20.720 chlorine results. It is not made quite clear in the text if this applies to the total chlorine as such, or the total *halogen* contents. If the latter, then the principle seems to be true, as there only exists a difference of 4 m.grms. per litre in what has been found and the above figure 20.720.

The mean of a number of chlorine determinations in the North and South Atlantic, respectively, give 20.3703

and 19.806 grms. Cl per litre from the *Challenger* samples. These numbers yield total solids 36.604 grms. and 35.765 grms. per litre. Therefore it would seem that the larger number of samples in the present case over the area yields a rather higher rate of total solids. The above figures were obtained from surface water, as were those of the present inquiry. An inspection of the tabulated results shows that there is an excess of chlorine—and, indirectly, of the total solids—in the North Atlantic. This result agrees with the *Challenger* figures above.

Solid Contents of the Ocean at approximately the Determined Locality.

Number of sample.	Latitude.	Longitude.	Contents per 1000 grms.	Remarks.
1	34°02 S	16°00 E	35.89	
2	27°15 S	11°09 E	35.63	
3	23°29 S	7°41 E	35.97	
4	19°42 S	4°16 E	36.19	
5	15°44 S	1°03 E	36.44	
6	11°43 S	2°05 W	36.15	
7	7°48 S	4°58 W	35.84	
8	3°53 S	7°50 W	35.59	
9	0°08 N	10°33 W	35.69	The Equator.
10	4°15 N	13°17 W	35.91	
11	8°15 N	15°42 W	34.51	{ During a continued heavy rain.
12	12°09 N	17°28 W	35.94	
13	16°26 N	17°49 W	35.83	
14	20°20 N	17°30 W	36.82	
15	24°22 N	16°55 W	36.69	
16	28°28 N	16°15 W	37.39	At Teneriffe; 200 yards from shore, in about 11 fathoms.
17	31°20 N	16°15 W	36.94	
18	34°48 N	15°34 W	36.85	
19	39°08 N	14°10 W	36.47	
20	43°11 N	12°50 W	36.39	
21	46°56 N	10°09 W	36.28	
22	English Channel		35.94	

The average sample yielded 36.31 total solids per 1000 grms. by multiplying the chlorine by 1.8058.

Carbon Dioxide.

Determinations of the alkalinity yield the proportion of CO₂ in the quantity tested. 250 c.c. of sea-water were boiled with an excess of N/10 HCl to a point short of dryness, and the volume restored with boiled CO₂-free distilled water. The excess of acid was titrated back with a corresponding solution of N/10 Na₂CO₃, the titre of which had been determined. By this method the *Challenger* samples yielded from 52 to 62.83 m.grms. CO₂ per litre. Estimations on the *Challenger*, made by Mr. Buchanan, gave 40 to 82 m.grms.

250 c.c. of the present sample were treated in a similar manner; this consequently yields the total CO₂. A second 250 c.c. was boiled down to free it from dissolved CO₂, made up with distilled water, acid added, and boiled down a second time. On making up the volume again with water, and titrating the excess acid in a similar manner, the small proportion of free dissolved CO₂ is calculated. These estimations were performed in duplicate, and gave—

Total CO ₂ per litre in m.grms. . . .	63.57
Free " " " " " " " " " "	5.11
Combined CO ₂ per litre	58.46

Sulphuric Acid.

The determination of this acid does not present any particular source of possible error. It was estimated in the usual manner as BaSO₄. The results were—

* " Report of Scientific Results of the Exploring Voyage of H.M.S. *Challenger*." London, 1884.

2.4681
2.4677

Mean, 2.4679 per 100 c.c.

Bromine.

The determination of this halogen was not conducted in the same manner as the *Challenger* samples were treated. Preference, for simplicity, was given to Fresenius's method. Finally, 100 c.c. were evaporated to dryness with a slight excess of pure solid sodic carbonate. The residue was extracted, after pulverisation, with four successive portions of absolute alcohol (boiling). The alcohol, after the addition of two drops of soda, was distilled off to dryness, the residue taken up with water and moderate excess of dilute nitric acid, filtered, and the solution precipitated with nitrate of silver. By this procedure, all the alkaline bromide is extracted, and only a small proportion of the chloride. The precipitate was dried and weighed, and the bulk of it removed to a hard glass bulb and chlorinated. From the loss in weight the bromide of silver is calculated and from it the bromine. The results of three estimations gave 67.10 m.grms. per litre; this is, by a singular coincidence, almost in absolute accord with the *Challenger* "surface" water figure of 67.04 per kilogram. Hence the present estimations appear slightly lower per kilo.

Lime.

CaO was estimated in 25 c.c. by volume in the usual manner, and as hereafter mentioned. In the first case, the base was precipitated as oxalate in the cold and allowed to remain overnight. The precipitate was washed by decantation, dissolved in dilute acid, and re-precipitated with ammonium oxalate and ammonia. It was finally weighed as CaO. In the second case, the volume to be examined was saturated with pure oxalic acid in slight excess and boiled. While hot, excess of strong ammonia (0.88) was added, and the mixture boiled for five minutes or until the precipitate became granular. This precipitate also was washed and re-precipitated, and finally weighed as CaO. All the lime estimations were multiplied by the factor 0.91 in the *Challenger* results as a correction for impurity. If this be applied to the present results, the percentage falls manifestly too low—therefore it has not been employed. The figures finally taken were—

0.06514
0.06520

Mean, 0.06518 CaO per cent (volume).

Magnesia.

MgO was determined in the filtrates from the two methods of lime estimation as pyrophosphate with a careful observation and exercise of all details to ensure its complete precipitation. By dissolving the weighed precipitates of pyrophosphate and searching for lime, no evidence of it could be found. The results are rather high when compared with the mean of the *Challenger* figures. If the amounts of CaO and SO₃ be relatively correct, it would almost appear as if the results were even below the truth. This is because the combined total CaSO₄ and MgSO₄ exceed rather more than is usual the total amount of the MgCl₂. The results were of MgO per 1000 c.c.—

2.4269
2.4273
2.4274
2.4267

Mean, 2.4270

Soda.

The *Challenger* procedure was adopted. A measured quantity of the sample was evaporated to dry-

ness with dilute sulphuric acid and ignited, to obtain a residue of normal sulphates. A specially constructed platinum vessel was employed for this in the *Challenger* estimations. The results nevertheless with an ordinary platinum crucible and lid gave constant residues. From the combined sulphates the found quantities of CaO, MgO, and K₂O expressed as sulphates were deducted. The remainder is normal Na₂SO₄, and is calculated into Na₂O or NaCl as desired. The found quantity of Na₂O is corrected by $\times 1.0043$, a factor used in the *Challenger* estimations, and from the corrected value the sodic chloride is determined. The results per litre were—

4.4571
4.4574
4.4587

Mean, 4.4577

Subtracting the found oxides as sulphates:—

CaO 0.06518 to CaSO₄ = 0.1583
MgO 0.24270 to MgSO₄ = 0.7281
K₂O 0.049952 to K₂SO₄ = 0.0924
0.9788

4.4577
0.9788

3.4789 Na₂SO₄

This obtained amount converted into the oxide and corrected gives 1.5266 Na₂O, and this is equivalent to 2.8971 NaCl per litre.

(To be continued).

NOTE ON THE VOLUMETRIC ESTIMATION OF CHROMIUM.

By RUDOLF L. LEFFLER, F.C.S.

A BROTHER chemist, estimating chromium according to Galbraith, expressed as his experience that he could, without transgressing the instructions, find that element in any class of steel or iron.

This peculiarity had been noticed in this laboratory fully eight years ago, and as the method seems to be still largely used and its reliability is open to question, I thought a short extract from a paper read by myself before the Sheffield Society of Engineers and Metallurgists, in March, 1895, might be of interest.

The original instructions consist in dissolving in dilute H₂SO₄ (volume not stated), oxidising the FeSO₄ to Fe₂(SO₄)₃ with KMnO₄, and the Cr₂O₃ to CrO₃, by adding as much again of that reagent as is required to oxidise the iron, filtering off the Mn precipitate, and titrating the chromic filtrate after the addition of ferrous salt with K₂Cr₂O₇.

It was brought to our notice when analysing very low chrome steels that our results were invariably too high, and on investigation it was found, if a sample of bar iron were treated precisely as if Cr were present, on titrating that the number of c.c. K₂Cr₂O₇ used was less than required by the ferrous salt added; this difference calculated out indicated a certain percentage of Cr. It then became customary to subtract this "blank" from our results. It was also found that the value of the "blank" varied with (1) the volume of acid taken to dissolve in (2) the excess of KMnO₄.

Influence of Acid.—Two grms. of steel, one free from and the other containing about 2 per cent, were dissolved in 100, 75, 50, and 30 c.c. respectively of H₂SO₄, boiled down till white sediment formed, taken up in boiling water, and finished in the usual way—with this exception, that only 0.125 gm. KMnO₄ was added in excess.

TABLE I.

Acid to dissolve.	Excess KMnO_4 .	Blank. P.c. Cr.	Steel.	Steel minus blank.
100 c.c.	0.125 grm.	0.08	2.15	2.07
75 "	"	0.06	2.13	2.07
50 "	"	0.05	2.08	2.03
30 "	"	Nil	2.04	2.04

Analysed by Stead's modified acid permanganate, hypo-aceto, and Arnold's phosphate, the average = 2.06 per cent Cr.

Influence of Excess KMnO_4 .—Two grms. of bar iron treated as in Table I.

TABLE II.

Acid to dissolve.	Excess KMnO_4 .	Blank, p.c. Cr.
30 c.c.	0.125 grm.	Nil
30 "	0.50 "	Nil
100 "	0.125 "	0.08
100 "	0.50 "	0.15
100 "	1.00 "	0.17

Next are given the results of a few chrome steels treated as in Table II.

TABLE III.

Mark.	Acid.	Excess KMnO_4 Grm.	Steel. blank.	Steel minus p.c. Cr.	P.c. Cr. in ppt.	Total p.c. Cr.	
M	30	0.125	2.19	2.19	Nil	2.19	Stead's acid
	30	0.50	2.15	2.15	0.03	2.18	Permanganate
	100	0.125	2.32	2.24	Nil	2.24	= 2.20
	100	0.50	2.31	2.16	0.04	2.20	
W	30	0.125	0.36	0.36	Nil	0.36	
	30	0.50	0.33	0.33	0.02	0.35	Do.
	100	0.125	0.43	0.35	Not done	0.35	= 0.37
	100	0.50	0.48	0.33	0.03	0.36	
H	100	0.50	2.21	2.06	0.02	2.08	Do.
	100	1.00	2.24	2.07	Not done	2.07	= 2.09
S	30	0.50	0.78	0.78	"	0.78	Do.
	100	0.50	0.91	0.76	"	0.76	= 0.78

The Cr in the precipitate was determined by dissolving in HCl, making up to 250 c.c., boiling till free from Cl, and titrating as usual.

Thus it is clearly seen how the method, in the hands of different chemists, may, through a lack of definite instruction, yield varying results—more particularly noticeable in low chrome steels.

It is probable that the "blank" is due to a reaction between the excess of acid and KMnO_4 , whereby some of the precipitate, which is somewhat soluble, is made active to ferrous salt. It was found on boiling some well washed precipitate in dilute acid, that the proportion thus active was small compared to the total amount dissolved.

The Laboratory,
Messrs. Thos. Firth and Sons, Limited,
Sheffield.

Preparation and Etherification of Dissymmetrical Dimethylsuccinic Acid.—E. E. Blaise.—The author finds that, in the case of succinic acid, the two carboxyls are not etherified simultaneously; that when the molecule is dissymmetrical, the two acid functions have very different values; the tertiary carboxyl being much more difficult to etherify than the primary. By this method it is very easy to decide whether the substituted succinic acid is symmetrical or not, and that there is not a constant relationship between the weights of the acid and neutral ethers formed at different moments of the reaction. —*Comptes Rendus*, cxvii, No. 10.

THE CYCLICAL LAW OF THE ELEMENTS.

By THOMAS BAYLEY, Assoc. R.C.Sc.

WHEN the elements are arranged in a line in the order of their atomic weights, it is apparent that there is from Li to F an orderly transition from the intensely positive alkali-metal type to the intensely negative halogen type, and then a sudden reappearance of the positive type in Na, after which there is a second orderly transition to the negative type (Cl), and then another change to positivity (K). Up to this point, therefore, two complete cyclical changes have been established, the first cycle from Li to Na—in all its essential details—being analogous with the second from Na to K. If then we proceed with the lineal arrangement, it is clear that we must first of all look for transitions and for reappearances of the alkali-metal type; in other words, for the continued development of the cyclical arrangement. And since analogous but modified transitions actually recur, and halogens followed by alkali-metals reappear, it is evident that the law of progression is actually cyclical. The first and fundamental feature of atomic progression is a progression in cycles.* The Li—Na cycle is a cycle involving the seven elements, Li, Be, B, C, N, O, and F, and the Na—K cycle involves also seven elements; and for this reason, and because in the succeeding cycles analogous groups of seven elements are repeated, almost all tabular expressions of the atomic progression have been set in a septenary form. But these arrangements, by unduly emphasising the septenary character of the periodic change, have perhaps somewhat obscured the more important and fundamental cyclical features of the progression. Before attempting to substantiate this assertion, however, it is necessary to consider the nature of the higher cycles which are complex, and to show that the properties of any element are more profoundly modified by position in the cycle than by position in the septenary series.

Assuming that hydrogen is the first element of an incompletely known cycle which we may call the first, then the Li—Na cycle becomes the second and the Na—K cycle becomes the third. Beyond this point the cycles are complex, and the fourth contains seventeen elements, of which the first, K, is completely analogous with Na; the second element, Ca, with Mg; and the third, Sc, with aluminium; for Na and K both conform to the alkali-metal type, Ca and Mg to the alkaline earth metal type, and Al and Sc to the type of the earth metals. But the analogies between Si and Ti, and between P and V, are less perfect, while the analogies between O and Cr and between Cl and Mn are very much less pronounced. The analogies between the latter pairs, indeed, appear to consist chiefly in structural resemblances between the compounds of the respective elements, due to the valency or atomicity impressed upon the septenary series as a primary law of its formation. The analogies between the elements of the septenary series entirely constituting the second and third simple cycles, and the elements constituting the first portion of the fourth cycle, a compound cycle, are perfect therefore only in the first, second, and third elements of each series respectively, and become fainter in the later members. Now the elements between which complete analogy exists are those which have analogous positions in the cycle, and the elements between which incomplete analogy exists are those which have not analogous cyclic position, but merely analogous serial position. The region of the third cycle—occupied by Na, Mg, and Al—corresponds to the region occupied by K, Ca, and Sc in the fourth cycle; for each group of elements occupies the front portion of its cycle, whereas Si, P, and S in the third cycle occupy a region posterior to the point of lowest atomic volume, while Ti, V, and Cr occupy a region anterior to the point of lowest atomic volume in the fourth. Neglecting for the moment the

* Bayley, *Phil. Mag.*, Jan., 1882.

position and properties of the three intermediate elements, Fe, Ni, and Co, and passing to the consideration of the second septenary series, which completes the fourth cycle, and to comparison of this with the third cycle, we see that analogy fails, or is chiefly structural, between Cu and Na, between Zn and Mg, between Ga and Al, but becomes more pronounced between Se and S and perfect between Br and Cl. The structurally analogous Cu and Na have nothing in common except serial position, and the fully analogous Br and Cl have similar cyclic position; Cl fails to find a complete analogue in Mn which has not analogous regional position, but finds it in Br, because both occur at the end of the cycle. To make this clearer it is necessary to study more closely the architecture of the simple and complex cycles. The increment of atomic weight occupied by the series Li—Na is 15.98, the increment from Na to K is 16.05, the increment from K to Fe is 16.86, the increment from Cu to Rb is 22, the increment from Rb to Ru is 16.2, and the increment from Ag to Cs is 25.04. The increments for the series constituting the simple cycles, and for the series occurring in the first portions of the fourth and fifth cycles, is therefore about 16 (or 17), and the increment for the series occurring in the posterior halves of the fourth and fifth cycles is 22 and 25. If we proceed to consider the nature of the sixth cycle, we are met at the outset by a difficulty, for since no alkali-metal of higher atomic weight than Cs has yet been discovered, it is not easy to fix the upper limit of the cycle.

The usual arrangement of the elements in odd and even series, such as Mendeleeff's original classification or Brauner's modification of it, show three septenary series and an intermediate group between Cs and Os, and the increment of 57 in atomic weight corresponding to this space is just about what would be required for 24 elements. Commencing with K and writing its higher analogues in a line, we obtain the sequence K, Cu, Rb, Ag, Cs, in which each alkali metal is followed by a sub-family analogue, and, unless other conditions intervene in the sixth cycle, the unknown element of atomic weight c. 156 should be an analogue of Cu and Ag, and the unknown element of atomic weight c. 170 should be an alkali metal and the first member of a seventh cycle. But as every alkali metal is preceded by a halogen (neglecting the argon family), and succeeded by an alkaline earth metal, it also follows that the element of atomic weight 170 should be similarly accompanied. Reasoning also from analogy, we must suppose that the unknown halogen is somewhat less negative than I, because negativity decreases with atomic weight in the halogen family, and the unknown alkali metal and the alkaline earth metal must be more positive than Cs and Ba respectively, because positivity increases with atomic weight in the highly positive families. It also seems necessary, from analogy, to assume the existence of an intermediate group between 152 and 154. But in all other instances, both lower and higher, in the atomic progression the intermediate groups are preceded not by basic earth metals (such as Sm), but by elements such as Cr and Mn, Mo and (—c. 100), and Ta and W, which form oxides of a pronounced acidic character. If, therefore, there is a cycle stretching from Cs to the position of atomic weight 170, it must differ essentially from compound cycles below it and the compound cycle above it. We may now profitably turn to a consideration of the curves of atomic volume in the various cycles. It was first pointed out by L. Meyer that atomic volume has a periodic variation, and the curves given in the accompanying figure are essentially identical with those drawn in his "Modern Theories of Chemistry."

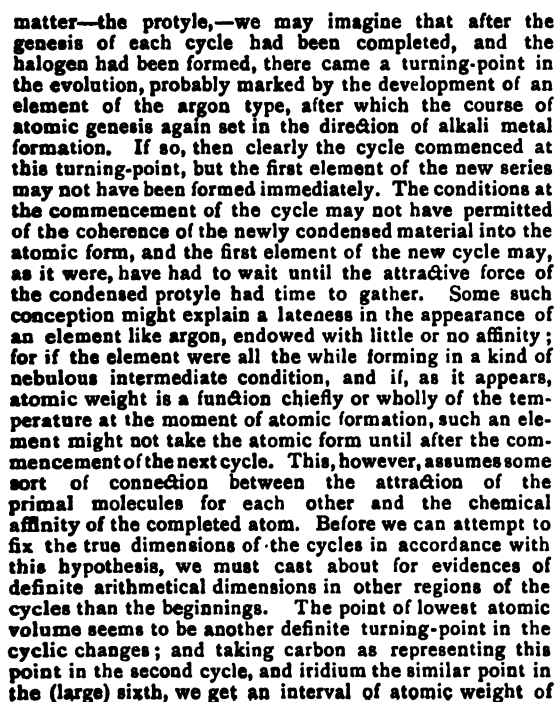
It is evident that atomic volume is a cyclical function. Each cycle, simple or compound, shows a loop, and all truly analogous elements occupy similar positions on the curves. The alkali metals have high atomic volumes, and occupy the top of the curves; the alkaline earth metals invariably occur on the downward flanks. The

earth metals occur lower down on the same flank, and in the fourth and fifth angles they immediately precede the elements (Ti, V, Cr, Mn), (Zr, Nb, Mo, —), which form acidic oxides. The intermediate groups occupy the troughs of the curves, and the series Cu—Br, Ag—I, Au—Bi occur on the rising posterior portion of the curves. If, then, the element of atomic weight c. 170 is an alkali metal, it is reasonable to conclude that it has a high atomic volume, and that the curve of atomic volume indicated by the upper dotted line must exist, even if it does not follow—however probable it may appear—that this curve need necessarily be as steep as it is drawn. The curves of atomic volume present very remarkable regularities, using the word curve to mean the collection of straight lines drawn from point to point. Thus the lines drawn from Cl to K, from Br to Ru, and from I to Cs, are almost exactly parallel, as are some other lines of similar character.

In the earlier diagrams, before the atomic volume of Cs was determined, the line from I to Cs was assumed to occupy a position in accordance with this parallelism, and the facts when determined justified the assumption, and for these reasons it seems permissible to use this method of drawing parallel lines as a means of forecasting the atomic volumes of elements as yet undiscovered. Now, whether we draw the upper or the lower dotted curve, in which latter case the metal (atomic weight c. 170) would not be a perfect analogue of Na and K, it appears necessary to believe in the existence of a halogen and an alkaline earth metal, and also of a group of intermediate elements of the iron or platinum type, followed by a series commencing with a true analogue of copper and silver. It is a significant fact that, although a number of elements are assigned to positions between atomic weight 150 and the atomic weights of Ta and W, there has as yet been described no element corresponding to the hypothetical intermediate group, or to the supposed halogen, alkali-metal, or alkaline earth metal. On the other hand, the numerous elements between atomic weight 150 and Ta which have been described are elements of a purely earth-forming type, separable from well-located earths, such as Sc, Yt, and La, which they closely resemble only by repeated fractional precipitations or similar methods. So far research has not rendered it possible to assign definite atomic weights to the majority of these rare earth metals, and much of the brilliant laborious work done upon them by Crookes and others has tended rather to bewilderment, yttrium and samarium, and several other rare earth metals, having been shown to be complex bodies; but accepting the usual location of these metals provisionally, it would seem that Tb > Er > Tm > Yb fall just where the hypothetical alkali-metal of atomic weight c. 170 would require to be placed, and that with these four elements there is a regular but small progression from greater to less basicity in the order given, which is what we might expect if the sixth cycle stretches from Cs to about atomic weight 233 with a long flank on the anterior (descending) portion of its curve of atomic volume occupied by a series of rare earth metals. This conception of the cyclic progression eliminates the unsymmetrical dotted curve, and substitutes a cycle of approximately symmetrical dimensions. The cyclical increments then appear as—

Cycle II.	Cycle III.	Cycle IV.	Cycle V.	Cycle VI.
c. 16	c. 16	46 (3 × 16)	47 (3 × 16)	94 (6 × 16)

and the position of lowest atomic volume, which in the lower cycles occurs near the middle of the curve, occupies in the sixth cycle a position nearer to the end. In these measurements of the length of the cycles, we have taken the increments of atomic weight from one alkali metal to the next higher in progression, but it does not necessarily follow that this is the proper method. For if the elements have been produced, as was suggested by Sir William Crookes, during the cooling of some primal form of


$$\begin{aligned} \text{Li } 7.01 + \frac{15.04}{3} &= 12.01 \text{ (C} = 11.97) \\ 22.05 + \frac{15.04}{3} &= 27.02 \text{ (Al} = 27.01) \\ 37.09 + \frac{15.04}{3} \times 4 &= 57.13 \text{ (Fe} = 55.9; \text{Ni} = 58.6) \end{aligned}$$

$$82.21 + \frac{15.04}{3} \times 4 = 102.25 \text{ (Ru} = 102.7)$$

$$127.33 + \frac{15.04}{3} \times 13 = 192.46 \text{ (Ir} = 192.5)$$

It may be assumed that the actual turning-point of lowest atomic volume may not be occupied by an element having the atomic weight corresponding to the temperature existing when the evolutionary crisis arrived.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, March 17th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

(Concluded from p. 150).

*38. "Contributions to the Chemistry of Thorium." By BOHUSLAV BRAUNER, Ph.D.

As there are few direct methods of separating the elements of the rare earths, the author investigated the reaction of Bahr (1864), who showed that thorium oxalate is easily soluble in a solution of ammonium oxalate. Bunsen (1876), though he found that the oxalates of the other rare earths are only slightly soluble in that reagent, had to repeat the process of solution many times to obtain a product which did not show in its spark spectrum the lines of other elements.

The reaction is due to the formation of a double thorium-ammonium oxalate, decomposed by water, and existing in solution only in the presence of free ammonium oxalate. After numerous crystallisations, a salt was obtained having the formula—



with one-third to one-half of free $(\text{NH}_4)_2\text{C}_2\text{O}_4 + \text{H}_2\text{O}$. Finding, however, that the solution readily becomes super-saturated, a compound having the formula—



was obtained in such a high state of purity that from its analysis the atomic weight of thorium, $\text{Th} = 232.59$, could be calculated.

This "complex" salt is ammonium thoroxalate. It is decomposed by water, but a definite quantity of the soluble product of decomposition dissolves the insoluble constituent, so that one part of water keeps one part of ammonium thoroxalate in solution if an additional half-molecule of free ammonium oxalate is present. The salt forms two hydrates, in accordance with Potilizin's law that a compound forming super-saturated solutions exists in several hydrate forms, one with $7\text{H}_2\text{O}$, the other with $4\text{H}_2\text{O}$. The former loses water in air of average humidity, passing into the latter form, which becomes anhydrous in perfectly dry air, or more readily at 100° , without loss of ammonia. On heating at higher temperatures, large quantities of cyanogen gas are evolved.

The amount of the decomposition of the salt caused by increasing the quantity of water was determined, together with the proportion of ammonium oxalate required to prevent decomposition. Three mols. of ammonium oxalate form, with 1 mol. of thorium oxalate, a clear solution in 300 mols. of water. This solution is, however, supersaturated with thorium oxalate, the latter separating until their relative proportions become 3.3 : 1. A convenient method for the purification of thorium may be based on these results, as the author has determined quantitatively the great difference in solubility between the oxalate of the feebly basic tetravalent thorium and those of the other trivalent rare earths in ammonium

oxalate. The rule was thus established: "the tendency to form complex oxalates is inversely proportional to the basicity of an earth." This, and another property of oxalates, that their stability under the oxidising action of nitric acid decreases considerably with an increasing basicity of the earth, was utilised for the purification of thorium.

On precipitating a solution of ammonium thoroxalate with oxalic acid, an acid thorium oxalate—thoroxalate of thorium and hydrogen—is formed, having the formula $\text{H}_2\text{Th}_2(\text{C}_2\text{O}_4)_5 + 9\text{H}_2\text{O}$. On using mineral acids (Glaser, *Zeit. Anal. Chem.*, 1897, xxxvi., 216; 1898, xxxvii., 25), a different result is observed.

*39. "On the Atomic Weight of Thorium." By BOHUSLAV BRAUNER, Ph.D.

The author determined the purity of the material, obtained, as described above, by the spark spectrum, and by atomic weight determinations. He determined in the air-dry oxalate (a) the thorium tetroxide by heating, and (b) the percentage of C_2O_3 by means of a solution of potassium permanganate. The first series of experiments gave concordant results, leading to the value $\text{Th} = 233.3$, a value not in agreement with that previously obtained by him, $\text{Th} = 232.59$. Further study showed that the oxalate used was contaminated with basic salt, and, a normal oxalate being prepared, the following values were obtained as a result of five series of experiments made with different preparations:— $\text{Th} = 232.50, 232.46, 232.45, 232.31, 232.33, 232.50, 232.44$, and 232.35 ; average, $\text{Th} = 232.42$.

This number agrees with that obtained by Krüss and Nilson, $\text{Th} = 232.45$. The author then investigates the conditions which led him to obtain the high value in the first series, approaching the value of Clève, $\text{Th} = 234.5$. The work of other investigators is vitiated by untrustworthy methods and impure material.

*40. "On the Compound Nature of Cerium." By BOHUSLAV BRAUNER, Ph.D.

The author showed in 1882 (*Trans.*, xli., 68; *Monats.*, iii., 486) that cerium from cerite is associated with an element whose higher oxide forms yellow and lower white salts, though the higher oxide is far less stable than cerium tetroxide. The substance is found with lanthanum and didymium when separating cerium by Bunsen's method. In 1885 the author showed (*Trans.*, xlvii., 879) that the substance remains in the mother-liquor after the bulk of the cerium sulphate has crystallised, and may be precipitated from it by alcohol. Schützenberger has repeated the author's experiments without mentioning his name. After applying Debray's method of separating cerium to these fractions and again fractionating, the author obtained substances with the following values for the atomic weights. The method employed was the analysis of the oxalate, and the determination of "active" ozonic oxygen by Bunsen's method in the oxide obtained by calcination, the latter number being given in parenthesis:— $\text{R}'' = 140.25 (4.64), 140.22 (4.65), 140.12 (4.81), 140.00 (4.65), 140.01 (4.59), 139.65 (4.61), 139.16 (4.01), 138.72 (4.51), 136.50 (3.31), 135.43 (3.93), 132.07 (3.73), 130.70 (3.21)$. As the value of the atomic weight decreases, the colour changes from white to a reddish brown orange.

The quantity of "active" oxygen, therefore, decreases almost in proportion to the decrease in value of the atomic weight. If we assume that the "active" oxygen is due entirely to the cerium present, the value of the atomic weight of the other element may be calculated at about 110. The author has thoroughly investigated the spark spectrum of the lower fractions, but has found no characteristic line other than those of cerium. In one case only were traces found of the two characteristic groups of lines in the red belonging to yttrium. No appreciable amount of yttria could be found, however, in the solution, even when a large quantity of the material in question was treated with potassium sulphate. As terbium forms a higher oxide of an orange colour, the colour of

the lower fractions of cerium may be due to its presence, but, as its atomic weight is higher than that of cerium, another earth of lower atomic weight must still be present, and it is not improbable, that, like gadolinium, this element may give no characteristic spectrum. The recent controversy between Wyruboff and Boudouard (*Comptes Rendus*, 1897, cxxv., *pass.*) has led the author to publish the result of this research, which has occupied him many years.

*41. "On Praseodidymium and Neodidymium." By BOHUSLAV BRAUNER, Ph.D.

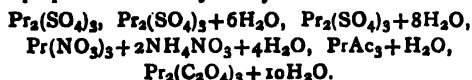
Applying Mendeleeff's method of crystallisation of the double nitrates with ammonium nitrate to a mixture of lanthanum and didymium, Auer von Welsbach (1885) succeeded in splitting up the old didymium into "praseodym" and "neodym." Praseodidymium, according to him, has an atomic weight $\text{Pr} = 143.6$, and forms two oxides, Pr_2O_3 and Pr_2O_5 . The higher oxide is black-brown, the lower forms green salts with a characteristic spectrum. Neodidymium, $\text{Nd} = 140.8$, gives only one oxide, Nd_2O_3 with pink salts possessing eleven absorption bands. Nothing of importance has been added to our knowledge on the subject during the last eleven years.

The author spent several years in repeating Welsbach's work, but last year he obtained from Dr. Waldron Shapleigh, in Gloucester, N.J., a quantity of highly purified research material.

From this mixture (40 grms. of praseodidymium oxide with 20 grms. of lanthanum oxide) the latter was removed, the former being converted by fusion with nitre into the oxide Pr_2O_5 . This was purified by fractionating with ammonium nitrate solution, ammonia, and oxalic acid.

A preliminary determination of the atomic weight gave the value $\text{Pr} = 140.8$. The analysis of the oxalate and the synthesis of the sulphate gave thirteen numbers, varying between 140.84 and 141.19, the average being $\text{Pr} = 140.95$. The lower oxide, Pr_2O_3 , is of a beautiful pale green colour, and the spectrum of its green salts contains the absorption bands $\lambda = 5968, 5895, 4812, 4693$, and 4447 (Rowl.). Praseodidymium trichloride gives a characteristic spark spectrum.

Salts of the trioxide having the following formulæ have been prepared and analysed by the author:—

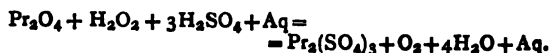


According to Professor Vrba's measurements, the salt $\text{Pr}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$ is isomorphous with $\text{Y}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$ and other sulphates of this type. This fact, and the slight solubility of the oxalate in ammonium oxalate, prove that the formula of the lower oxide is Pr_2O_3 , and not Pr_2O_4 or Pr_2O_5 , in which cases the atomic weight would be 188 or 235. The tetroxide, Pr_2O_4 , obtained from the nitrate on heating to a temperature of 440° , or by fusion with nitrate at 400° , is jet-black, but it becomes dark brown in a state of fine division.

Up to the present, only its basic salts have been obtained; the sulphate $2\text{Pr}_2\text{O}_4 \cdot \text{SO}_3 + 2\frac{1}{2}\text{H}_2\text{O}$, and the basic acetate in acetic acid solution, having a formula which may be represented by either $\text{Ac}_3\text{—PrIV O—PrIV—}(\text{OH})_3$, or $\text{Ac}_2\text{—PrIII}_2\text{O}_2\text{—OH.Ac} + \text{H}_2\text{O}$. The double fluorides are being investigated.

It is of the greatest importance to know whether the tetroxide is (a) a true oxide of the water type, with ozonic oxygen (like lead) tetroxide, PbO_2 , in which case the praseodidymium would be tetravalent, or (b) a true peroxide of the hydrogen peroxide type, with antozonic oxygen (like barium peroxide, BaO_2), in which case the metal would be trivalent. With regard to the first view, it yields, with dilute acids, free oxygen, and the solution does not reduce potassium permanganate (negative test for hydrogen peroxide). On treatment with concentrated nitric and sulphuric acids, oxygen containing much ozone

is evolved, and with hydrochloric acid chlorine is set free, arising probably from the decomposition of the unstable perchloride, PrCl_4 (?). Like other ozonic oxides of the water type, it presents with hydrogen peroxide the phenomenon of catalysis in the presence of dilute sulphuric acid, a quantity of the hydrogen peroxide being oxidised, strictly equivalent to the "active" oxygen lost by praseodidymium tetroxide according to the equation—



On the other hand, praseodidymium tetroxide gives with ether, water, sulphuric acid, and potassium bichromate an intensely blue colouration (Baresville's reaction) according to the equation—



In the treatment, therefore, of the tetroxide with dilute sulphuric acid, both reactions take place simultaneously, and the result is the evolution of oxygen,—



Praseodidymium tetroxide is, therefore, an oxide of a new kind, belonging simultaneously to the ozonic oxides of the water type, and to the antozonic oxides of the hydrogen peroxide type; it is, in fact, the missing link between these two hitherto entirely different types of peroxides, its active oxygen being at the same time both entirely ozonic and entirely antozonic.

The basic acetate of the tetroxide, which is nearly white, and in the spectrum of which the blue absorption bands are invisible when seen by reflected light, though the yellow band remains unchanged (an indication of the complex nature of praseodidymium), is the salt of an oxide belonging to the hydrogen peroxide type, for it reduces permanganate quantitatively, and gives Baresville's reaction for hydrogen peroxide. It is, however, so remarkably stable that it does not part with all its active oxygen, even after being boiled for an hour with concentrated caustic potash solution.

As regards the author's neodidymium, its absorption spectrum contains the bands $\lambda = 7283, 7080, 6905, 6368, 6279, 6247, 5788, 5323, 5211, 5097, 5063$, and 4280. It contains, in addition to these, a strong sharp band of wave-length $\lambda = 4694$, which differs in character and position from the praseodidymium band $\lambda = 4693$. There were also traces of $\text{Pr } 4812$ and $\text{Pr } 4447$, $\text{Dy } 4752$ and 4605 (Sm. series?).

The value of the atomic weight of neodidymium found after the first purification of the material by treatment with oxalic acid was $\text{Nd} = 143.4$, after another purification $\text{Nd} = 143.63$. Its lower oxide, having the formula Nd_2O_3 , is of a beautiful pink colour with an amethyst tint, whereas the higher oxide contains a little more oxygen than corresponds to the formula Nd_2O_5 (the author's old D_2O_5). Neodidymium also gives two series of salts, the acetate of the higher oxide being nearly white. According to a quantitative analysis, by comparing the intensity of the bands the author's neodidymium contained 2.9 per cent praseodidymium, whereas the old didymium from cerite contained in 100 parts of the oxide 78.8 parts Nd_2O_3 and only 21.2 per cent Pr_2O_3 .

With regard to the position of these elements in the periodic system, the author concludes, from the tendency of them both to become more highly oxidised than would correspond to the formulæ Pr_2O_4 and Nd_2O_5 , that praseodidymium and neodidymium may be further split up. This is regarded as very probable by all rare earth chemists. The pure oxides will probably be found to have the formulæ Pr_2O_5 and Nd_2O_6 , so that the eighth series of the periodic system would assume the following form:—

I. Ca.	II. Ba	III. La.	IV. Ce.	V. Pr.	VI. Nd.
133	137.4	138.2	139.7	141	143.6.

DISCUSSION.

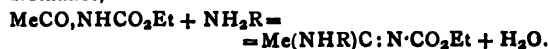
The PRESIDENT congratulated Professor Brauner on the valuable scientific results he had presented to the Society, and on behalf of the members expressed their appreciation of the motives which had induced the author to come all the way from Prague to communicate four papers of such great importance to the Chemical Society. He wished him success in the continuation of such laborious and intricate investigation.

42. "Action of Ammonia and Substituted Ammonias on Acetylurethane." By GEORGE YOUNG, Ph.D., and ERNEST CLARK.

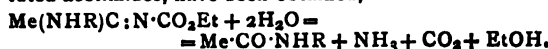
Acetylurethane has been subjected to the action of ammonia and of substituted ammonias under varying conditions as to solvent, temperature, and pressure. The general results of the investigation show that action takes place principally according to the equation—



Under certain conditions the products point to the action having resulted in the formation of acetamidine urethanes,—



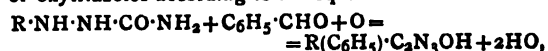
These acetamidines have not been isolated, but in their place the products of hydrolysis, acetamide and substituted acetimides, have been obtained,—



Ammonia and methylamine enter into action most easily, piperidine, aniline, the naphthylamines, and phenylurea less so. Acetylurea, acetanilide, and diphenylamine appear to be without action.

43. "Formation of Oxytriazoles from Semicarbazides." By GEORGE YOUNG, Ph.D., and BENJAMIN MITCHELL STOCKWELL, B.Sc.

This paper contains an account of the formation of of oxytriazoles according to the equation—



where R is an aromatic radicle. The following substances are described:—*Paratolylsemicarbazide*, m. p. 187–188°. *Acetylparatolylsemicarbazide*, m. p. 212.5°. *Benzoylparatolylsemicarbazide*, m. p. 218°. *Paratolylazocarbamide*, m. p. 142°. *5-Phenyl-1-paratolyl-3-oxytriazole*, m. p. 242°. *Acetylphenylparatolyl-oxytriazole*, m. p. 112–113°. *Benzoylphenylparatolyl-oxytriazole*, m. p. 132°. *Phenylparatolylthioxytriazole*, m. p. 51–52°. *β-Naphthylsemicarbazide*, m. p. 225°. *Phenyl-β-naphthyl-oxytriazole*, m. p. 274–275°. *Acetylphenyl-β-naphthyl-oxytriazole*, m. p. 142–143°. *Benzoylphenyl-β-naphthyl-oxytriazole*, m. p. 141–142°. *Metanitrophenylsemicarbazide*, m. p. 195°. *Benzoylmetanitrophenylsemicarbazide*, m. p. 188–189°. *Metanitrophenylazocarbamide*, m. p. 168–169°. *5-Phenyl-1-metanitrophenyl-3-oxytriazole*, m. p. 235°. *Acetylphenyl-metanitrophenyl-oxytriazole*, m. p. 130–132°. *Benzoyl-metanitrodiphenyl-oxytriazole*, m. p. 168°. *Nitrodiphenylthioxytriazole*, m. p. 96°.

44. "Formation of *aa*-Dihydroxypyridine." By S. RUHEMANN, Ph.D., M.A.

Ethyl *aa*-dihydroxydinicotinate (*Proc.*, 1898, xiv., 47) when boiled with concentrated hydrochloric acid, yields the hydrochloride of *aa*-dihydroxypyridine, from which ammonia sets free the base m. p. 192–193°. The same substance is obtained from Guthzeit and Dressel's monethylic ethoxy-pyridone dicarboxylate (*Annalen*, 1891, cclxii., 113) by boiling hydrochloric acid.

45. "Position-isomerism and Optical Activity; the Comparative Rotatory Powers of Diethylic Mono-benzoyl and Mono-toluyll Tartrates." By PERCY FRANKLAND, F.R.S., and J. MCCRAE, Ph.D.

The authors review the present state of knowledge

concerning the relative rotatory powers of the phenyl and three toluyll derivatives of optically active compounds, and point out that, whilst of the three isomeric toluyll groups the ortho- has almost invariably the least and the para- the greatest rotatory influence, the rotatory influence of the phenyl group is in some series greater, and in some less, than that of any one or of all of the toluyll groups.

As a contribution to this study, the authors have prepared the compounds mentioned above, and have determined their rotations in a fused state over a wide range of temperature, whilst they have also determined the molecular weight and rotation of each in glacial acetic acid solution, with a view of ascertaining the relative rotations of the compounds in the monomolecular condition.

The following are the specific rotations at 100° for each of the compounds, as well as of diethylic tartrate, from which they may be regarded as derived.

Diethylic tartrate	$[\alpha]_D 20^\circ = +7.66^\circ$	$[\alpha]_D 100^\circ = +15.77^\circ$
Diethylic mono-benzoyltartrate	$[\alpha]_D 24^\circ = +20.71^\circ$	$[\alpha]_{99.5^\circ} = +17.69^\circ$
Diethylic mono- <i>o</i> -toluylltartrate	$[\alpha]_D 20^\circ = +11.82^\circ$	$[\alpha]_D 100^\circ = +10.88^\circ$
Diethylic mono- <i>m</i> -toluylltartrate	$[\alpha]_D 20^\circ = +13.59^\circ$	$[\alpha]_D 100^\circ = +12.57^\circ$
Diethylic mono- <i>p</i> -toluylltartrate		$[\alpha]_D 100^\circ = +15.85^\circ$

Thus the dextrorotation of diethylic tartrate increases with rise of temperature, whilst that of the above monoacyl tartrates diminishes. In glacial acetic acid solution, the benzoyltartrate has a lower rotation than the para-toluylltartrate, but otherwise the above order of the rotations is unchanged. In all cases the rotation in glacial acetic acid solution was considerably lower than that of the pure substance, but this difference is not attributable to the pure substance in the fused state being dissociated, for the rotation of the diethylic monobenzoyltartrate in benzene solution was even lower still, and yet in benzene solution it is more probable that there would be association than in glacial acetic acid. Thus from the results in benzene it would appear that association of the benzoyl compound is attended with diminution in the rotation, so that the higher rotation of the liquid benzoyl compound itself cannot be due to association also. Neither do the molecular volumes of these compounds point to association when interpreted by Traube's formula.

Diethylic monobenzoyltartrate has been previously prepared by Guye and Fayollat (*Bull. Soc. Chim.*, 1895, [3], xiii., 201); their preparation was admittedly impure, and yielded a very much lower rotation and melting-point. It was first prepared by Perkin (*Trans.*, 1867, xx., 138), but without determining the rotation, the melting-point which he obtained was almost exactly the same as that found by the authors.

46. "The Action of Di-isocyanates upon Amido-compounds." By H. LLOYD SNAPE, D.Sc., Ph.D.

By the action of diphenylene di-isocyanate upon phenylhydrazin in ethereal solution, *diphenylenediphenylsemicarbazide*,—

$\text{Ph}\cdot\text{NH}\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}\cdot\text{C}_6\text{H}_4\cdot\text{C}_6\text{H}_4\cdot\text{NH}\cdot\text{CO}\cdot\text{NH}\cdot\text{NH}\cdot\text{Ph}$, was obtained. The product was a white powder which was insoluble in the more common organic solvents. It gave with copper sulphate a chocolate-brown colour, which was changed to green on the addition of ammonia.

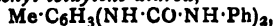
Toluylenediphenylsemicarbazide,—



(1 : 2 : 4) was prepared by acting with 1 : 2 : 4-toluylene-di-isocyanate on an ethereal solution of phenylhydrazin. The product, after re-crystallisation from alcohol, consisted of colourless crystals which decomposed and rose in a capillary tube at 203°. This carbazide also was insoluble in the more common organic solvents, with the

exception of alcohol, in which it was difficultly soluble. With copper sulphate, it gave a wine-red colouration which changed to green on the addition of ammonia.

1:2:4-Diphenyl-toluylen-diurea,—



was obtained on mixing ethereal solutions of aniline and toluylenediisocyanate. The product was re-crystallised from alcohol; it formed microscopic needles which melted at 261° with accompanying decomposition. It was difficultly soluble in methyl, ethyl, and amyl alcohol.

1:2:4-Toluylen-diurea, $\text{Me} \cdot \text{C}_6\text{H}_3(\text{NH} \cdot \text{CO} \cdot \text{NH}_2)_2$, was prepared by treating an ethereal solution of toluylenediisocyanate with ammonia and crystallising the product from water. Minute crystals, which melted with accompanying decomposition at about 252° . It was sparingly soluble in alcohol. Lussy had previously described this reaction, but states that the product melts at 220° .

47. "The Action of Alkyl Iodides on Silver Malate and on Silver Lactate." By THOMAS PURDIE, F.R.S., and G. DRUCE LANDER, B.Sc.

The object of this research was to discover the cause of the abnormally high optical activity of the ethereal malates and lactates prepared by the silver salt method. An abstract of the first part of the paper treating of the action of isopropyl iodide and ethyl iodide on silver malate has already been published as a preliminary note (*Proc.*, 1896, xii., 219). The high activity of the malates, thus prepared, is due to the simultaneous production of ethereal salts of the much more active alkyloxysuccinic acids.

The silver salt of the inactive lactic acid acts in a similar manner. With isopropyl iodide, the ethereal salt of (*m*- or *iso*-) propoxypropionic acid is produced in considerable quantity. By reactions similar to those employed in the case of silver malate, the substance was separated from the ethereal lactate with which it is mixed, and identified by the analysis of several salts obtained from it. Evidence was also obtained that with ethyl iodide the ethylic salt of ethoxypropionic acid is produced, though in this case, as in the corresponding reaction with silver malate, it was not possible to isolate the salts in the pure state owing to the small quantity of the alkyloxy-acid formed. The authors have recently effected the resolution of several alkyloxy-propionic acids by means of alkaloids, and find that these acids are highly active as compared with lactic acid. Their production in the reaction above referred to accounts therefore for the abnormal activity of the ethereal lactates prepared from silver lactate.

The reaction seems to be a general one for the silver salts of hydroxy-acids (see following paper), and cannot therefore be relied on for the preparation of the ethereal salts of these acids in the pure state. With the permission of Mr. Brame, the authors have recently extended his experiments on the action of alkyl iodides on silver tartrate. The results already obtained point to the formation of dialkyloxysuccinates being the cause of the abnormally high activity of the ethereal tartrates thus prepared. The authors are carrying on the research with the view of throwing light on the reaction by which the alkyloxy-acids are produced, and of adapting it, if possible, for the direct preparation of optically active acids of this kind from the corresponding active hydroxy acids.

48. "On the Optical Rotations of Methyl and Ethyl Tartrates." By J. W. RODGER and J. S. S. BRAME.

The authors, in attempting to prepare the alkyl tartrates in a high state of purity, find that when prepared by saturating an alcoholic solution of the acid with hydrochloric acid, or heating the acid or mono-substituted ester with alcohol in sealed tubes, the rotation of the product is much lower than that obtained for esters prepared by the action of an alkyl iodide on silver tartrate.

Differences of the same nature have been observed by J. Wallace Walker (*Trans.*, 1895, lxvii., 914) in the case of the lactates, and by Purdie and Williamson (*Trans.*,

1896, lxix., 818) for malates and lactates. With alkyl tartrates, however, the difference in activity is much greater than with lactates or malates. Further, the rotation for different specimens of tartrates prepared by the silver method is by no means constant, although every precaution was taken to ensure similar treatment during preparation.

In order to determine any other differences in the esters prepared by these different methods, samples of methyl tartrate giving these different optical activities were hydrolysed with excess of sodium hydrate, when the products of hydrolysis gave practically the same rotations. Secondly, no difference in the refractive indices was found; and on combustion of methyl and ethyl tartrates prepared by different methods and varying widely in rotation, no real difference in the percentage of carbon could be detected.

These abnormal results for the rotations may be explained on three hypotheses. The low rotation of the ethereal salts prepared by hydrochloric acid saturation or by the sealed tube method may be due to racemisation. Second, the esters from the silver salts may be isomeric with and more active than those prepared by other methods; or third, the esters prepared from silver salts may be contaminated with some much more optically active substance.

The first hypothesis is precluded by the constancy of the rotations obtained by the authors for specimens prepared by three different methods and the agreement with the rotations observed by others with these substances. The second is improbable, but receives some support from the result of hydrolysis. With regard to the third hypothesis, Purdie and Lander in a preliminary note on the action of alkyl iodides on silver malate (*Proc.*, 1896, xii., 221) ascribe the higher rotation given by the malates so obtained to the presence of "small quantities of the ethereal salts of the highly active alkyloxy-acids." The same explanation may hold with the tartrates, in which case these derivatives must be highly active, for they can only be present in small amount, since there is no appreciable rise in the percentage of carbon found. If the high rotation for tartrates prepared from silver salts is due to this cause, then the agreement of results obtained on hydrolysis is only a coincidence.

NOTICES OF BOOKS.

Agriculture in some of its Relations with Chemistry.

By F. H. STORER, S.B., A.M., Professor of Agricultural Chemistry in Harvard University. In three volumes. Seventh Edition, revised and enlarged. London: Sampson Low, Marston, and Co. 1897. Pp. 1901.

AGRICULTURE being perhaps the most important industry in the world, for "The farmer feeds them all," it is with pleasure that we welcome another, the seventh edition of Professor Storer's valuable work. It differs from the previous editions in that it is printed in larger type, and necessarily from new plates; it has been entirely re-written, and a large amount of fresh matter has been interpolated.

Vol. I. is divided into thirteen chapters; the first four deal with the general relations of the soil, air, and water to plant life, and to each other, water being a subject of great importance as the means of bringing the plant-food to the plant, though some leaves can absorb ammonia from the air; the amount of ammonia present, however, is so slight that it cannot have much effect on the nourishment of the plant. Very striking results are said to have been obtained by placing lumps of solid carbonate of ammonium on the hot water pipes of a conservatory, so that the amount of ammonia in the air was increased to from 2 to 4 parts per 10,000 by weight.

The characters of different soils and their tillage occupy chapters v. and vi., after which we come to manures, soils considered as chemical agents, and modes of action of special manures, such as gypsum or plaster-of-Paris, which is described as capricious and uncertain. It is recommended to be strewn with a liberal hand on moist places in stables and cow-stalls, as it checks the fermentation of the urine, thought it should not be added directly to the manure heap. Chapter x. deals with phosphatic fertilisers, and chapters xi., xii., and xiii., with the various assimilable nitrogen compounds.

Passing to Vol. II., which contains fourteen chapters, we begin with the question of nitrogenised animal and vegetable refuse, or the less easily assimilable kind; farm yard manure is of this class, and the effect of it is felt for three years, though two consecutive crops of wheat will exhaust it.

We now come, in chapter xv., to an entirely different subject, namely, symbiosis or blended growth. It is one of the commonest things in nature, whether in animal, fish, reptile, bird, or plant life, to find one species living with another. In the case of plants they are generally parasites. In this chapter we also find an interesting description of the root-nodules, which have recently been proved to fix nitrogen from the air and supply it to the plants. In chapter xvi. we read about the various conditions and functions of carbonic acid, and in chapter xvii. about green-manuring, the use of sea-weed as manure, &c. Humus or vegetable mould is a reservoir of nitrogen; it resists decay, but little is known as yet as to its precise chemical composition. Its value as a manure depends upon several properties. First, no doubt, its power of supplying nitrogen to the plants; then because of its porosity it imbibes and holds water, by its lightness it improves many soils, it promotes chemical action in the soil both by means of the acids it contains and by those caused by its eventual decay. Chap. xxi. deals with the different methods of applying manure; the best method naturally seems to be to place it deep enough to get the capillary moisture, but not so deep that the air cannot gain access to it. In chap. xxiv. we come to potassic manures. It has already been shown that potash is essential for the life of plants; in fact, in ancient times potash was known as the "vegetable alkali" to distinguish it from soda, which was called the "mineral alkali." A great deal of the potash in soils comes from the decomposition of felspar; nearly all clays and clayey soils contain potash, and much is returned to the land from the crops, but the waste must be made up by potash salts of some kind, according to the kind of crops; for instance, chloride of potassium will not do for tobacco, as chlorides hinder its burning. There is a good deal of difference of opinion as to the use of lime as a manure. This matter, and a great deal more, bearing on the same subject is fully discussed in chap. xxvi.

Vol. iii. also contains fourteen chapters, the first of which, chap. xxviii., is historical and reviews the history of manures. Next come the theory of the rotation of crops, and special systems of rotation. One striking illustration of the significance of rotation is afforded by the so-called fairy-rings in old grass fields; they are really circles of grass of coarser growth and greener colour than the rest, and are caused by the growth of a fungus from a spore which happened to fall there; it grows, dies, and decays, and thus furnishes extra nourishment to the grass; after a time a new crop of fungus grows from the spores shed by the first lot, and so it gradually spreads.

The practice of rotation is not based upon chemical or botanical considerations, but is of great antiquity. In England and Germany, however, it took its rise from the village common-land system, where to avoid disputes the land was marked off into three great parts, one for winter grain, one for summer grain, while the third was left fallow; then each year (like Alice's tea-party) they shifted round one.

The important questions of irrigation and sewage are

dealt with in chapters xxxii. and xxxiii. Historians can tell us that civilisation had its rise in irrigated countries. In Egypt, for instance, where we are in the habit of saying man first became civilised; though we think it highly probable that man had already been civilised long before in some other place. Peru, also a rainless country, was skillfully irrigated by the Incas. Strange to say, the art of irrigation suffered an unaccountable decline, and the result was always the downfall of the nation. Irrigation keeps the land sweet, and this is one of its principal merits.

A great deal of useful and interesting information is to be found in chap. xxv., on generalities as to the growth of crops. We then come to several chapters devoted specifically to barley, oats, and hay, and the establishment and maintenance of hayfields; and in chap. xxxix. we have the theory and practice of making hay. Turning the hay frequently prevents fermentation and waste.

The last chapter of this comprehensive work deals with ensilage. In recent years it has been found to be a great improvement to build the silo in the form of a high bin, in or near the barn, instead of digging holes in the ground as formerly practised. One difficulty in ensilage is to check fermentation: M. Mueller made a very successful experiment of using bisulphide of carbon as a germicide; a lot of grass which had begun to decay, together with a lot of buckwheat, sunflower plants, cabbage leaves, tops of beets, and some straw and chaff, were siloed, with bisulphide of carbon sprinkled on between the layers; when the silo was opened the fodder was found to be a compact, well-preserved mass, readily and greedily eaten by cattle, and all smell of the bisulphide had disappeared.

At the end of Vol. III. we find a copious and excellent index to all three volumes, and we must finally express our admiration for the excellent arrangement and masterly handling of the whole subject.

CHEMICAL NOTICES FROM FOREIGN SOURCES

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 10, March 7, 1898.

Chemical Actions of the Electric Discharge. The Aldehyde and Nitrogen.—M. Berthelot.—Continuing his experiments on the chemical action of the electric discharge, the author found it necessary to follow the comparative study of the fixation of the nitrogen in the aldehydic compounds. He gives a long list of experiments tried and results obtained with:—A. *Aldehyds of the Fatty Series*: (1) Ethylic aldehyd, C_2H_4O ; (2) Primary propylic aldehyd, C_3H_6O ; (3) Acetone, or secondary propylic aldehyd, C_3H_6O ; (4) Dimethylic methylate, $C_3H_8O_2$. B. *Derivatives from the Aldehyds of the Fatty Series*: (1) Aldol, $C_4H_8O_2$; (2) Paraldehyd, $C_6H_{12}O_3$; (3) Trioxymethylene, $C_3H_6O_3$; (4) A 60 per cent solution of CH_2O . C. *Aldehyds belonging to a less Hydrogenised and Cyclic Series*: (1) Camphor, $C_{10}H_{16}O$; (2) Benzylic aldehyd, C_7H_6O ; (3) Benzoin, $C_{12}H_{12}O_2$; (4) Cinnamic aldehyd, C_9H_8O ; (5) Salicylic aldehyd, $C_7H_6O_2$; (6) Furfural, $C_5H_4O_2$; (7) Glucose, $C_6H_{12}O_6 \cdot H_2O$, and its derivatives. As a result of this large number of experiments, the author finds that, under the influence of the electric discharge, all the aldehyds examined fix nitrogen, forming condensed compounds, amides or amines; the fixation of the nitrogen does not cause so great a loss of hydrogen as in the case of carbides and the corresponding alcohols.

Chemical Action of the Electric Discharge. Organic Acids and Nitrogen.—M. Berthelot.—In this

paper the author describes his experiments on:—A. *The Monobasic Acids*, such as acetic, propionic, formic, &c. B. *The Monobasic Acids with a Simple Function*: Crotonic and benzoic acids. C. *Bibasic Acids with a Simple Function*, such as succinic, fumaric, phthalic, &c. D. *The Alcohol Acids*, lactic, malic, &c. E. *Acids with a Complex Function*, pyruvic, levulic, &c. He finds that, as a rule, the organic acids fix nitrogen in the same manner as the alcohols and the aldehydes. This fixation, in the case of acids, occurs without any loss of hydrogen. In the case of acetic and propionic acids, one molecule of acid fixed an atom and a half of nitrogen; the limit with higher acids has not been determined. No oxygen is lost in either the solid or liquid compounds.

Observations Relative to the Chemical Action of the Electric Discharge on Dielectric Liquids.—M. Berthelot.—The author has continued his experiments to liquids, taking care to control the strength of the current so as to prevent the formation of sparks in the liquid. He has tried turpentine, olive oil, and absolute alcohol. His results show that the discharge acts on organic liquids as on gases, causing polymerisations and the separation of hydrogen, but the action is much slower, because of the low conductivity and the great mobility of the liquids.

Action of Sulphate of Lime on some Alkaline Haloid Salts.—A. Ditte.—Not suitable for abstraction.

Preparation of Glucinum by Electrolysis.—P. Lebeau.—Will be inserted in full.

Chemical Estimation of the Carbonic Acid in the Air, even when only Present in Traces.—M. Nicloux.—The method used, which is described at length, makes it easy to estimate very accurately the amount of carbonic oxide present in the air, in proportions varying between 1/100,000 and 1/50,000th.

The Dissociation of Carbides of Barium and Manganese.—MM. Gin and Leleux.—By using a very large current in the electric furnace, viz., 1600 amperes and 35 volts, the authors have succeeded in dissociating the carbides of barium and manganese and volatilising the metal, leaving a deposit of coke at the bottom of the crucible. In the case of the manganese, the characteristic reddish fumes due to the oxidation of the metallic vapour on contact with the air are easily observed.

Synthetic Isoborneols: their Identity with the Phenolic Alcohols.—G. Bouchardat and J. Lafont.—Not suitable for abstraction.

On the Partial Decomposition of Chloroform in an Organism.—A. Desgrez and M. Nicloux.—In some experiments with dogs the authors found that the quantity of carbonic acid in the blood increased in one case from 1.6 c.c. per litre to 2.9 c.c. after three hours ten minutes of anaesthesia: similar experiments on larger dogs showed even a greater difference. The authors therefore state that these experiments confirm certain conclusions already arrived at by them by means of their fire-damp meter—that chloroform is decomposed in the animal economy with formation of carbonic oxide.

Berichte der Deutschen Chemischen Gesellschaft.
1898, No. 2.

Decomposition of Wool Fat.—L. Darmsteadter and J. Lifschütz.—The fat was decomposed, after the wax had been separated, and an analysis made of the various acids and alcohols.

New Synthesis of Adenin and its Derivatives.—Emil Fischer.—Amido-oxychlorpurin is converted into adenin by the action of phosphorus oxychloride, which yields dichloradenin. This latter, on reduction with hydriodic acid, gives adenin. Methyl-derivatives of adenin are similarly prepared from methylamido-oxychlorpurin.

A Fluosilicate and a Fluophosphate of Potassium and Rubidium.—R. F. Weinland and J. Alfa.—These salts are obtained by acting with HF on the sulphate and phosphate of potassium. Fluosulphate, $\text{K}_2\text{O}_7\text{F}_2\text{HK}_2\text{H}_2\text{O}$, separates from a solution of neutral K_2SO_4 in a 40 per cent solution of HF in a crystalline form after concentrating by evaporation, and gives a good yield. The Rb salt is formed in the same way.

Formation and Change of Hylotropisomeric Substances.—Karl Schaum.

Separation of Mercury and Bismuth Salts.—L. Varino and F. Treubert.—A weak solution of mercuric chloride and bismuth oxychloride in HCl is decomposed with a mixture of hypophosphorous acid and hydrogen peroxide; the mercury is obtained quantitatively as calomel, whilst the bismuth remains in solution.

Molecular Weight of Solid Bodies.—J. Traube.—The author shows that the molecular co-volume of a solid is approximately half that determined for liquids in a previous paper. This proves an aggregation of molecules in pairs in the solid state. These results were proved experimentally for a large number of solid organic bodies. A similar property is shown also for many inorganic salts, which, as a rule, are bi-molecular aggregates. However, when in solution they give three ions; they are, for the most part, mono-molecular.

Some Thiourea Derivatives.—Rudolf Andreasch.—From thiourea the author prepared thiohydantoin and a great many derivatives (ethyl thiohydantoin, ethylphenylthiohydantoin, &c.) as crystalline compounds. He was also successful in preparing from thiourea, thioparabamic acid, and parabamic acid. This reaction consisted in treating thiourea with cyanogen and subsequent hydrolysis.

Copper Compounds of Dicarboxylglutaconic Ester.—Wilhelm Wislicenus.—The author succeeded in preparing copper compounds of dicarboxylglutaconic ester by treating the latter with the calculated amount of copper acetate. Two such copper compounds were prepared; one neutral and one basic, which latter could be converted into the neutral by further treatment with dicarboxylglutaconic ester.

Condensation-products of Aldehyds with Phenol and Phenolcarboxylic Acids, and the Diphenylmethane Colour Derivatives of these.—Kahl Leopold.

Ammonium Peroxide.—P. Melikoff and L. Pissarsjewsky.—If an ethereal solution of H_2O_2 is added to a large excess of an ethereal solution of ammonia, the substances being cooled with calcium chloride and snow, a solid compact crystalline substance is obtained. The crystals, after washing with cold ether, were dried on a tile and were analysed. They appeared to correspond to the formula $(\text{NH}_4)_2\text{O}_2 + \text{H}_2\text{O}_2 + \text{H}_2\text{O}$. At ordinary temperatures the substance decomposed, first dissociating into NH_3 and H_2O_2 , and then giving off oxygen energetically, and forming a small quantity of ammonium nitrate.

Osmotic Pressure and Electrolytic Dissociation.—J. Traube.—The author determines the osmotic pressure of bodies in solution from a knowledge of the co-volume, which latter depends only on the concentration of the solution, and is independent of the nature of the dissolved substance.

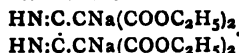
Some Mono- and Di-halogen Ketones.—Franz Kunckell and Friedrich Johannsen.

Some Condensation-products of Piperonal, Vanillin, and Protocatechuic Aldehyds.—M. Rogon.

The so-called Nitramine and Isonitramine and their Ethers.—A. Hantzsch.—The author describes several nitramine and isonitramine compounds of the aromatic series, and shows the difference exhibited in their chemical and physical properties.

Diazo-compounds of *m*-Phenylendiamine.—K. Eiermann.—The compounds formed by this reaction are diacetyltriarnidoazobenzene and triarnidoazobenzene. The former was diazotised again, and after treatment with more phenylendiamine, yielded a diazo-product of the latter; this compound has been known as Bismarck brown, though its constitution has not hitherto been proved.

Addition of Cyanogen to Sodium Salt of Malonic Acid.—Wilhelm Traube.—An alcoholic solution of acid sodium malonic ether absorbs cyanogen when it is heated. The liquid becomes intensely red, and after a short time a dark red crystalline precipitate comes down. The analysis of this product shows it to have the composition—



Some Brominated Ketones.—Franz Kunckell and Wilhelm Scheven.—A number of these substances were prepared from bromacetyl bromide on phenol and its ether.

Homologues of Oxalacetic Esters.—Wilhelm Wislicenus and Max Kiesewelter.

Hydrazinacetic Acids.—Wilhelm Traube.—This substance, the amido-glycolol, was obtained in crystalline form from oxybenzylhydrazinacetic acid on hydrolysis. The ethyl ester of this acid was prepared; also the ethyl ester of amidohydrantoinic acid and amidohydrantoin itself.

The Proteolytic Enzyme of the Saps of Yeast.—M. Hahm.

Separation of the two Desmotropic Forms of Acetoacetic Esters.—Robert Schiff.

Alcoholic Fermentations without Yeast.—Eduard Buchner and Rudolf Rapp.

Aliphatic Nitroso-compounds.—O. Piloty and O. Ruff.

MISCELLANEOUS.

Royal Institution of Great Britain.—The following arrangements have been made for the Friday Evening Lectures after Easter:—

April 22. W. H. M. Christie, C.B., M.A., F.R.S., Astronomer Royal, "The Recent Eclipse."

April 29. Professor Andrew Gray, M.A., LL.D., F.R.S., "Magneto-optic Rotation and its Explanation by a Gyrostatic Medium." (With Experimental Illustrations).

May 6. Edward A. Minchin, M.A., Fellow of Merton College, Oxford, "Living Crystals."

May 13. Professor W. A. Tilden, D.Sc., F.R.S., "Recent Experiments on certain of the Chemical Elements in relation to Heat."

May 20. The Right Hon. D. H. Madden, LL.D., "The Early Life and Work of Shakespeare."

May 27. Lieut.-General The Hon. Sir Andrew Clarke, R.E., G.C.M.G., "Sir Stamford Raffles and the Malay States."

June 3. Professor W. M. Flinders Petrie, D.C.L., "The Development of the Tomb in Egypt."

June 10. The Right Hon. Lord Rayleigh, M.A., D.C.L., LL.D., F.R.S.

Other Lectures will be delivered as follows, on the dates mentioned, at 3 o'clock precisely:—

Tuesdays, April 19, 26. Two Lectures on "Phases of Art—Past and Present," by Thomas Cooper Gotch.

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CONTENTS.

ARTICLES :—	PAGE
Bakerian Lecture.—Further Experiments on the Action exerted by certain Metals and other Bodies on a Photographic Plate, by W. J. Russell, Ph.D., V.P.R.S.	167
On the Composition of the Atlantic Ocean, by C. J. S. Makin, F.I.C., F.C.S.	171
On the Preparation of Glucinum by Electrolysis, by P. Lebeau	173
Note on the Separation and Estimation of Lead, Copper, and Arsenic, by F. Jean	173
A Simple and Accurate Method of Testing Diastatic Substances, by Jokichi Takamine	173
The Dignity of Analytical Work, by C. B. Dudley	173
PROCEEDINGS OF SOCIETIES—	
CHEMICAL SOCIETY.	173
NOTICES OF BOOKS.—The Calorific Power of Fuels, founded on Scheurer-Kestner's "Pouvoir Calorifique des Combustibles"—Quantitative Chemical Analysis by Electrolysis.	176
CHEMICAL NOTICES FROM FOREIGN SOURCES	177
MEETINGS FOR THE WEEK	177

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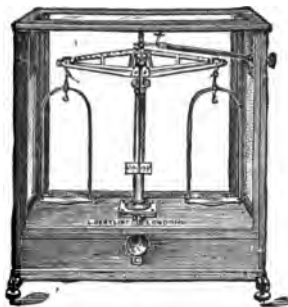
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BAKERIAN LECTURE.

FURTHER EXPERIMENTS ON THE ACTION EXERTED BY CERTAIN METALS AND OTHER BODIES ON A PHOTOGRAPHIC PLATE.*

By W. J. RUSSELL, Ph.D., V.P.R.S.

In a paper read before this Society in June last (*Roy. Soc. Proc.*, lxi., p. 424; *CHEM. NEWS*, lxxv., p. 302) it was stated that certain metals, alloys, and other substances,—such as picture copal, printing ink, straw board, &c.,—were able to act even at a distance on a sensitive photographic plate, producing effects similar in appearance and developed in the same way as plates which had been acted on by ordinary light. At that time sufficient experimental evidence had not been obtained to determine the nature of this action, or even to clearly indicate its general character, whether in fact the action rose from vapour given off by the active body, or whether phosphorescence was produced which acted on the plate. That bodies so slightly volatile as zinc, aluminium, nickel, &c., should be able to give off at ordinary temperatures in a few days sufficient vapour to act strongly on a photographic plate, and that such vapour should be able to pass rapidly through media, such as gelatin, celluloid, collodion, &c., seemed difficult to realise, although many of the earlier experiments appeared to indicate that this was the kind of action which took place. Later experiments confirm the view that a vapour is given off, which is the cause of the action on the plate.

Certain organic bodies, as well as metals, have been shown to act on the photographic plate, and in endeavouring to ascertain the nature of this action experiments with organic bodies were first undertaken, as the results which they yield are more easily and rapidly obtained than those with the metals, and if their mode of action was determined it would probably throw light on the action exerted by the metals. In the former communication it was stated that printing ink and copal varnish are active substances, both when in direct contact with a photographic plate and when at a distance from it. Further, it was found that the action which they exerted was able to pass through different media. Although printing inks and copal varnishes may vary considerably in composition, the main constituents are constant,—hence it was easy to determine that boiled oil and turpentine were the bodies to which they owed their activity, and that these bodies separately behaved in the same way as did printing ink and copal varnish. Boiled oil—that is, linseed oil which has been heated with oxide of lead—is an active substance, and most of the following experiments have been made with the pale drying oil which is prepared by Messrs. Winsor and Newton. Pure turpentine is also a very active substance, and, owing to its volatility, in many experiments very useful. These bodies can be used either as liquids in small dishes or by saturating Bristol board or other neutral and porous bodies, such as ignited pumice stone, &c., with them. In the case of the drying oil it can be painted on glass or cardboard and allowed to harden.

The experiments described in the former communication have been repeated under the same and under slightly different conditions and in another laboratory; the results

obtained, with one exception, confirm the previous statements. Glass, selenite, mica, even in very thin layers, are absolutely opaque to the action, whereas gelatin, celluloid, collodion, gutta-percha, tissue tracing-paper, parchment, and paper are more or less transparent. Linseed oil and turpentine may fairly be taken as typical examples of active organic bodies. This property of acting on the photographic plate is far from belonging to all volatile organic bodies; for instance, although vegetable oils have the power of acting, mineral oils, so far as they have been tried, have not the power. Benzene, carbon disulphide, chloroform, &c., also are without this power of acting on the photographic plate; but the question of what substances are and what are not active will be dealt with on another occasion; at present it is simply to consider the conditions under which linseed oil and turpentine act on the photographic plate. The picture copal varnish which was much used in the former experiments obviously owes its activity to the turpentine and the oil it contains. Warm the varnish for some length of time; these bodies are driven off, and an inactive gum remains. This experiment obviously suggests a vapour as the cause of the action; at the same time such a vapour would pass through bodies such as gelatin, celluloid, &c. With regard, for instance, to the thin sheet gelatin, it appears to offer but very slight obstruction to the action of these organic bodies; if the thickness of the gelatin be increased still the action takes place, only the time of exposure has to be considerably longer.* Another striking fact with regard to this emanation from these active bodies is that it gives an accurate picture of the surface from which it has come. A hard copal surface on glass will give a picture showing every brush mark, unevenness, and scratch on the surface, and if the action take place through a thin sheet of gelatin, or even as many as six or more sheets, still the picture of the scratches is distinct. The following experiments are apparently strong evidence that the action on the photographic plate is due to the vapour given off from these organic bodies.

A piece of Bristol board saturated with drying oil, or a piece of glass painted with it or with picture copal, is placed on the bottom of an ordinary plate box, and a photographic plate larger than the active body is suspended above it with the film upwards; light is excluded from the box, and the arrangement is left for, say, a fortnight; then the plate when developed will be found to have been acted on irregularly round its edge, at some parts considerably more than at others, but everywhere shading off and evidently in the way, which would occur if a vapour had rolled round the edge. Another experiment which showed this kind of action very satisfactorily was carried out as follows:—A circular piece of the Bristol board was saturated with drying oil, and at a little distance above it a smaller circle of mica, which is perfectly opaque to the action, was placed, and again above this was another piece of mica with a circular hole smaller than the circular mica plate, and then the photographic plate was placed above. By this arrangement no direct action could take place between the drying oil and the sensitive plate, but a vapour could work its way between the mica plates, and thus reach the photographic plate; and this it did, for after an exposure of three days, on developing the plate there was a dark ring formed shading off towards the centre. Another and very simple experiment illustrating this same point is to place a small circular glass dish, with some drying oil in it, in the middle of a photographic plate, and leave it there for a week. On developing the plate it will be found that no action has taken place where the dish stood, but that immediately beyond the outside of the dish much action has occurred, and that the darkening gradually fades away. There is still another way in which the action of

* A Lecture delivered at the Royal Society, March 24th, 1898.

* The thin gelatin is 0.02 m.m. and the thick 0.16 m.m. in thickness.

these organic bodies has been tested, and that is by transferring the active power of these bodies to a neutral substance. If vapour be the immediate cause of the darkening of the photographic plate, then it would be possible, if a piece of Bristol board were suspended above drying oil, for instance, for the inactive board to take up those vapours and become photographically active. This was found to take place. Bristol board of good quality is a very useful substance in all these experiments, both as a screen and as an absorbent. It is in itself an inactive body, and may be heated in a water-bath before using, to prevent the accidental presence of any substance which might act on the plate. If a piece of the Bristol board be suspended above drying oil, in the liquid or solid state, or turpentine or picture copal for two or three days, or even less, it becomes strongly active, and when placed in contact with a photographic plate quickly darkens it. This action of the Bristol board is well shown, if a pattern be stamped upon it, which is easily done by pressing against it a piece of white net; black net must not be used, as it is slightly active; then the charged Bristol board will give an unmistakable picture. If turpentine be the active substance used to charge the Bristol board the exposure need only be for a few hours, but if after this charging it be exposed to the air for a day or two its activity will be found to have gone. There is obviously no visible indication of this activity of the Bristol board, and consequently if a device be cut out on a screen which is placed in front of a sheet of cardboard, or any inactive paper, and it is exposed to turpentine or to oil, or if the vapour of these bodies be in any other way brought in contact with the paper, a dark picture of such device, which is not visible, may be produced. Some unexpected and curious cases of ghostly pictures thus formed have been met with; they are, however, all produced in this way, and need not be described now. The above experiments have been made at ordinary temperatures, but if the temperature be increased the activity of these organic bodies is also greatly increased. High temperatures cannot, of course, be used, but a temperature of 55° C. does not appear to alter the photographic plate. With drying oil—which is one of the most active substances that has been used—it is easy to obtain a picture in thirty minutes at the above temperature. The interesting pictures which in the former communication it was shown could be produced simply by laying a piece of dry wood or the section of the branch of a tree on a plate are produced by the volatile matter contained in the wood. These pictures appear at first sight very accurate and complete; but this is not really so, for some parts of the structure of the wood is not shown and other parts are too strongly developed, depending on the amount of volatile substance present in the different parts. It is, however, very remarkable that so small a quantity of the volatile body as exists in a piece of dry wood should be able to produce a picture: the activity of the wood is increased by the presence of moisture. This property of acting on the photographic plate, possessed by the linseed oil, belongs apparently to the vapour and not to the oil itself; for if a sheet of thin gelatin be placed on a photographic plate, and on it a thick glass ring nearly filled with oil, and over the top of the ring another sheet of gelatin, not in contact with the oil, and another sensitive plate, it will be found that after a week's exposure no action has taken place below the oil, but that a large amount has occurred above it where the vapour has penetrated the upper sheet of gelatin. A similar result is also obtained by simply floating a piece of the thin gelatin on a dish of oil and placing a sensitive plate above. At the sides where the vapour can form and get away there is action on the plate; in the centre there is none.

In addition to glass, mica, and bodies of that kind, the action does not take place through a layer, except it be very thin, of either gum arabic or of paraffin. If a piece of Bristol board or a glass plate has hardened

drying oil on it, and be painted over with a strong solution of gum arabic which is allowed to dry, then the delicate cracking which occurs can be very well shown on a photographic plate.

Pure water does not act on the plate, neither does pure alcohol or pure ether, but the ordinary commercial specimens of the last two bodies do, and often to a considerable extent. Alcohol which produces a dark picture will, after digestion with lime and careful distillation, be entirely inactive, and ether after careful purification also becomes inactive. Moisture, if present, does not effect this result; thus the presence of certain impurities, and they appear to be some of the most common ones, can be readily photographed, and approximately their amount determined by the darkness of the picture formed, so that by this means can be determined whether, for instance, a purification process is working well and whether it has completely done its work. The pictures are easily obtained by placing some of the liquid to be tested in a small glass dish, and a sensitive plate above it. Obviously it is only certain impurities which will be indicated in this way, but the reaction is otherwise of considerable importance, for it gives a simple method of determining what bodies soluble in these liquids are capable of acting on a sensitive plate. This matter will be treated of in a separate communication.

That the vapour given off by these organic bodies is the immediate cause of the action on the sensitive plate the above experiments seem to show. At the same time it is remarkable that such a vapour should readily pass through media such as gelatin, celluloid, &c., and not by mere absorption, but in such a way as to produce a picture of the surface from which it emanated.

Passing on to the action which certain metals exert on a photographic plate, results have been obtained which are strikingly similar to those just described. Substitute a piece of polished zinc for a piece of Bristol board saturated with linseed oil, and similar effects are produced on a photographic plate; the time of exposure must, however, be longer. Although both magnesium and cadmium are slightly more active than zinc, this last metal is the most convenient one to experiment with, and has been used in most of the following experiments. In addition to the above three metals, nickel, aluminium, lead, and bismuth have the same property, but not so strongly developed, of acting, both when in contact and when at a distance on a photographic plate. Cobalt, tin, and antimony can also act in the same way, but their action is considerably feeble, and undoubtedly other metals can act in the same way, but require much longer exposure. Mercury, which in the former paper was stated to be the most active of all the metals which had been tried, is now found to be quite inactive; the metal used in the former experiments was impure. This matter will be explained further on.

As so strong a similarity exists between the effects produced by the above-mentioned organic bodies and the metals, the question which naturally presents itself is, do they also give off a vapour which directly or indirectly acts on the photographic plate? The following experiments show that such an action does probably occur. Zinc which has been long exposed to the air is inactive. An exposure out of doors for only three or four days diminishes very considerably its activity, but covered up in doors after three weeks it will still give a tolerably dark picture. If it has a bright but perfectly smooth surface it is active, but not strongly so; rub the zinc with coarse sand or emery paper, and it is then obtained in its state of greatest activity; the same applies to all the metals. If, when in this condition, any of the active metals be placed in contact with a photographic plate, a beautifully sharp picture of the scratched surface is obtained. The great increase of the fresh metallic surface produced by the rubbing may account for the increase of activity which the scratching produces. If the zinc plate be raised only slightly above the photographic plate, a

sharp picture of the scratches is still obtained, and of course as the distance is increased, so is the indistinctness of the picture increased, and at last it fades into a general cloudiness, and in this form the zinc plate can easily be made to act through the distance of an inch or more.

This action of the metals passes through the same media as do the vapours from the organic bodies, and clear pictures can be obtained through sheets of thin gelatin, &c.; in fact, what has already been said with regard to the transmission of the activity of the organic bodies applies to the metals; gelatin, both thin and thick, allows the action of the metal to pass through it; celluloid and collodion do the same, and so does gold-beaters' skin and tracing paper. Reasoning, then, from this strong analogy between the action of the organic and the metallic bodies, it must be assumed that the above-mentioned metals from a clean surface and at ordinary temperatures give off vapour, and this vapour apparently acts when under the same circumstances in a like manner to the vapour given off by the drying oil. It gives a clear picture of the metal surface from which it arose, and it can permeate the same media as the organic vapours. The remarkably clear pictures of, for instance, a zinc surface, which can be produced through a sheet, or even several sheets, of the thin gelatin proves that the action is not one of mere absorption.

To gain further knowledge on this point and test the porosity of these different media, the power of hydrogen to diffuse through them was tried by cementing specimens of the different substances on glass tubes, which were filled with hydrogen and placed over water. The action is somewhat remarkable, but requires further confirmation. With the thin gelatin ordinary diffusion does not occur, and a hardly, if any, perceptible rise of the water in the tube occurs on starting the experiment; but on standing for some length of time—two or three days—the water begins to rise, and after a week or more it will stand at a height of four or more inches above the level in the vessel, and there it remains at least for a month or more. With the thick gelatin there is no evidence of any diffusion occurring. Celluloid acts much in the same way as the thin gelatin, a column of water gradually rises and remains there. The action of the gutta-percha tissue is to absorb the hydrogen; the diffusion tube completely fills up with water, and remains full without showing any tendency to fall for a couple of months, and then the experiment was stopped. With tracing paper diffusion occurs in the ordinary manner, and the same happened in the single experiment tried with gold-beaters' skin. That the rise of the water in the diffusion tubes is not owing to a mere absorption of the hydrogen by the gelatin or gutta-percha has been proved by placing a considerable quantity of these bodies in a tube sealed up at one end, filled with hydrogen and inverted over water; after several weeks no rise of the water in the tube occurred. The above experiments have been repeated with the same results, but further trials are being made. Possibly the metallic vapour is in a still finer molecular state than ordinary hydrogen, and thus is able easily to permeate a medium which hydrogen can only slowly get through, and air cannot get through. At all events, this may be looked upon for the moment as a working hypothesis.

That the action of the metals like that of the organic bodies is due to a vapour can be demonstrated by experiments exactly similar to those already described. For instance, if the thin mica plates be arranged above a zinc plate, in the way already mentioned, so as to cut off all direct action between the zinc and sensitive plate, a ring of action is produced which can only be accounted for by supposing a vapour present, which has worked its way between the sheets of mica and thus gained access to the photographic plate. Again, a piece of Bristol board can be made active by contact with, or mere proximity to, a piece of polished zinc. A striking instance of this arose in the following way:—A piece of perforated zinc had lain on the bottom of an ordinary plate box for a con-

siderable length of time, probably about two months; the zinc was then taken away, and a sensitive plate dropped into its place. On developing this plate, a picture of the perforated zinc was obtained. Other experiments of a similar kind have been made. If the Bristol board be not in direct contact with the zinc; if a screen, with holes cut out in it, be interposed, it will be found that the Bristol board where exposed to the direct action of the zinc will become active, and will give an exact picture of the holes or whatever design it may be which has been cut out on the screen. To produce this effect the cardboard has to be exposed to the zinc for fully six weeks. This changing of the Bristol board does not take place satisfactorily above ordinary temperatures. With other metals than zinc, these changing effects have not as yet been obtained.

Another experiment, which illustrates the way in which the metals can act, is to take a piece of ordinary perforated zinc, polish one side, and lay this polished side against a plate of plain glass in a printing frame, then place the photographic plate against the dull side of the perforated zinc, and leave it in the dark for three or four days; then, on developing the plate, a reversed picture is obtained,—that is, the holes in the zinc will be represented by dark spaces, and the zinc itself by light ones. If the holes in the perforated zinc are large they are represented by shaded circles, so that these pictures are produced by the vapour emitted by the polished zinc which has crept into the open spaces and thus gained access to the photographic plate. It has already been shown that the action exerted by zinc passes more readily down a glass than down a paper tube of the same size: this has been strikingly confirmed by taking two pieces of glass tubing 1 inch long, $\frac{3}{8}$ inch in diameter; inside one a single coil of inactive paper was placed, and both tubes stood on a sheet of polished zinc, and a photographic plate rested on the top of them. They were then left for a week, and on developing the plate a black patch appeared above the tube without the paper, and no action was visible above the one with the paper. Without removing the paper, it was painted over with melted paraffin, and again a photographic plate put on the top of the two tubes; now two circular dark patches were produced of equal intensity.

If the activity of the zinc depends on a vapour which it emits, it seemed possible that it could be carried along by a stream of air. In order to try whether this was the case, a tube a foot long was packed with zinc turnings which had been amalgamated, and a stream of pure air sent through it. The end of the tube was fixed into the side of a dark box and a sensitive plate with a screen upon it suspended above it; thus no direct action could be exerted by the amalgamated zinc on the plate. The experiment was continued for four days, then, on developing the plate, a picture of the screen above where the tube entered the box was obtained, but at the other end of the plate there was no action.

The presence of mercury in this experiment was unsatisfactory, and might account for the result obtained, therefore an exactly similar experiment was made, and zinc turnings alone were used and a plate without a screen. The experiment was carried on for a week, and it was then found that a black patch had been produced immediately above the end of the tube. To be sure that this darkening did not arise from the action of the air, the whole of the zinc was removed from the tube and again the air sent through it for a week; the sensitive plate showed no signs of any action having taken place.

To try whether any accumulation of the vapour, and hence an increase of action, could be brought about by an increase of zinc surface, two small circular glass dishes were taken, about $1\frac{1}{2}$ inches in diameter and $\frac{1}{4}$ inch in depth; into one a single disc of bright zinc foil was placed, and in the other twenty similar discs, then on the top of both vessels a photographic plate was placed. The single disc was raised on a piece of glass, so both end

discs were at the same distance from the plate. The discs were a little smaller than the glass vessels, and, owing to their not being quite flat, there was a space between each one. In two experiments there was no marked difference between the density of the pictures produced, the single disc produced as much effect as the twenty. A more direct way for the passing off of the vapour was then made by cutting a circular hole $\frac{3}{8}$ inch in diameter through the centre of all the zinc discs, and now a very black central spot was formed by the twenty zincs, and of course there was a white spot with the single disc, so that the vapour accumulated to a very considerable extent in this central space.

It has already been mentioned that the statement in the former paper that mercury was the most active of the metals is incorrect. The error arose from not suspecting that a trace of any impurity would affect the activity of the mercury, and, consequently, not taking special precautions to insure its perfect purity. On repeating the former experiments with another sample of mercury it was found that no action occurred, which seemed very remarkable; moisture was added, the temperature was increased, but still no action took place; the addition of a little zinc to the mercury was then tried, and it was found that this made the mercury excessively active. The presence of a very small quantity of zinc is able to effect this change, certainly less than $\frac{1}{3000}$ per cent. It is very remarkable that so small an amount of the metal can cause so strong an action on the photographic plate, for the exposure to the vapour given off by such an amalgam need not, even at ordinary temperature, be longer than two to three days. If other active metals are dissolved in pure mercury they act in the same way; at all events, this applies to magnesium and to lead. If silver, on the contrary, be added it does not render mercury active, nor does sodium. This action of mercury, which contains zinc or lead, the most common impurities, is so readily recognised that it becomes a valuable test for its purity, and a very interesting means of following the effect produced by any purifying process. A specimen of mercury containing not more than $\frac{1}{3000}$ per cent of zinc gave a very dark picture; this mercury was then treated first with sulphuric acid and afterwards, for three days, with nitric acid, and the picture it then gave was very faint, and on repeating this purifying process no picture at all was produced. Again, a sample of mercury containing zinc was carefully distilled. The distillate gave a very faint but very distinct picture. Another sample of distilled mercury also gave a faint picture.

Temperature, as might be expected, affects greatly this activity of the metals; at 4° or 5° C. zinc has but little action on a photographic plate. Most of the foregoing experiments have been made at about 17° or 18° C., and some, as specially noted, at 55° C. The Ilford special rapid plates have been used.

It appears, then, from the above experiments that certain metals have the property of giving off, even at ordinary temperatures, vapour which affects a sensitive photographic plate, that this vapour can be carried along by a current of air, and that it has the power of passing through thin sheets of such bodies as gelatin, celluloid, collodion, &c.; in fact, so transparent are these bodies to the vapour that, even after it has passed through them, it is able to produce clear pictures of the surface of the metal from which it came. That much remains to discover with regard to this action of the metals is obvious, the most active metals are not the most volatile. Nickel is very active, cobalt only very slightly so, copper and iron are practically inactive. I hope before long to be able to bring before the Society further developments of these curious actions, both of metals and organic bodies.

The foregoing experiments have been made in the Davy-Faraday laboratory, and I beg to thank the managers of the Royal Institution for having allowed me to work in their laboratory. My thanks are also due to my assistant,

Mr. Block, for the very careful and intelligent way in which he has aided me with the experiments.

March 24, 1898.—Additional experiments have been made with active organic substances, in order to determine to what class of bodies they belong. As already stated, linseed oil and turpentine were the two substances first found to be very active organic bodies, and following out these results it has been proved that vegetable oils in general have more or less this property of acting on a photographic plate. Linseed oil, for instance, is very active, olive oil only very slightly so. Even samples of "pure" linseed oil obtained from the different artists' colourmen vary considerably in the amount of action which they exert. The boiled, or drying linseed oil, has, at ordinary temperatures, the same activity as the ordinary oil, but under other conditions it appears there is some difference in their action. Another class of bodies, also called oils, namely the essential oils, have been found to be very active substances. Samples of commercial specimens of the following essential oils have been tried:—Peppermint, lemons, pine, juniper, bergamot, winter green, lavender, cloves, eucalyptus, cadjeput, and cedrat, and were all found to be active, and not only when used by themselves, but also when dissolved in a large amount of pure alcohol. It is well known that the active substances in the essential oils are bodies known as terpenes, and I have to thank Dr. Tilden for supplying me with pure specimens of these bodies; they are all of them remarkably active. Paraldehyde and benzaldehyde are also very active bodies. Ordinary aldehyde and formaldehyde, as they have been at present applied, are only slightly active. Guaiacum, both when in powder and in alcoholic solution, is active, and so are powdered cinnamon, sweet spirits of nitre, and eau de cologne. Brandy is slightly so. Now the important property belonging to all these bodies is their reducing or oxygen-absorbing power, hence the conclusion that it is this property which enables them to act on the photographic plate. The mineral oils, purified petroleum spirit, alcohol, ether, the esters, such as ethyl acetate, benzene, nitrobenzene, are all, when pure, unable to produce any effect on a sensitive plate, and even oxidised bodies nearly related to the terpenes, such as terpinol and camphor, are not active, neither is thymol. Terebene is an exceedingly active body. The difference of activity of the ordinary oils seems to follow their oxygen-absorbing power, at all events it is so with regard to linseed and olive oils, for the former is the most active of the oils, and 1 grm. of it is said to be capable of absorbing 186 c.c. of oxygen, while the same weight of olive oil can absorb only 8.2 c.c. It is also interesting to note that at least with some of these active bodies results can be obtained which correspond to what photographers term solarisation or reversal, the action, when modified, giving a black picture, when carried to its full extent a white one.

It has already been stated that a mere trace of zinc makes mercury active, but it is certainly equally curious and unexpected that a trace of zinc should make alcohol active. It is only necessary to take pure alcohol and place in it some strips of bright zinc foil, leaving the metal in for three or four days. It will then be found that the alcohol can act strongly on a photographic plate. The same happens with ether and with ethylacetate, but not with benzene. In addition to zinc, cadmium, magnesium, aluminium, and fusible metal can act in the same way, whereas lead, nickel, tin, silver, sodium, and, as far as experiments have gone, all the inactive metals, have no such power. This reaction is still being investigated, but certain it is that careful filtration does not remove this activity from alcohol, nor does distillation entirely destroy it.

ON THE
COMPOSITION OF THE ATLANTIC OCEAN.

By C. J. S. MAKIN, F.I.C., F.C.S.
(Concluded from p. 156).

Potash.

THIS oxide is undoubtedly the most difficult to determine with accuracy. The conventional method of separation and weighing as K_2PtCl_6 was proved impracticable in such an enormous excess of soda. The present estimations were carried out in accordance first with the *Challenger* method and then with two modifications. One of the latter has been adopted. In the first, the sulphates obtained in the Na_2SO_4 estimation were dissolved in a little water and then rather more than 1½ parts of $PtCl_4$ added for every part of K_2SO_4 present. The mixture was evaporated short of dryness, and, on cooling, a fair quantity of a mixture of 2 parts of absolute alcohol and 1 part of ether added, and the mixture left for several hours. The precipitate was washed by decantation with a similar mixture of alcohol and ether, and then removed to a small filter and the washing proceeded with until the wash-water is quite colourless and a few c.c. of it are free from solid matter on evaporation. The precipitate is dried and reduced in a porcelain basin by a stream of hydrogen; heat up to 300° being employed. The residue is washed with water and dilute hydrochloric acid, and the platinum finally weighed. This weight multiplied by 0.4747 equals K_2O in sample taken. In the *Challenger* estimations, the proportions of potash were fairly constant, and it was found that 100 c.c. of water usually yielded about 100 m.grms. of platinum.

The Second Method.

Twenty-five c.c. of the original sea-water were rendered faintly acid and mixed with $PtCl_4$ in sufficient excess, and evaporated to a syrup without previous elimination of sulphuric acid. When cooled, 90 per cent alcohol was added, and, after the mixture had stood some hours, a little ether was poured into the basin. The precipitate, collected on a small filter, was washed until the filtrate was quite colourless, dissolved in boiling water, and poured in this condition into a boiling solution of formate of soda. The boiling was gently prolonged for five minutes, the precipitated platinum filtered off and weighed, and the same factor used as in the first estimations.

The Third Method.

The alkaline chlorides were carefully isolated from 25 c.c. of the sea-water and treated as in the last stage. The first precipitate of K_2PtCl_6 obtained was, however, dissolved in a very little boiling water, and the solution evaporated a second time with the addition of a little more $PtCl_4$. Subsequent treatment was similar to the last method.

The results were rather conflicting.

The first gave	103.70 m.grms. platinum
	103.61 " "
The second gave	97.67 " "
	98.04 " "
The third gave	105.20 " "
	105.26 " "

The mean of the third was adopted. This figure (105.23) yields of K_2O 0.049952, or 0.07915 KCl.

Ammonia.

These determinations were made as follows:—100 c.c. of the sample and 400 c.c. of re-distilled ammonia-free water were distilled and nesslerised in the conventional manner. The bulk of the residual liquid was restored to 500 c.c. and excess of potash added. Distillation was then continued, and the ammonia estimated and returned as "ammoniacal salts." Permanganate of potash was then added, the volume restored to 500 c.c., and distilled.

tion proceeded with; this yields albumenoid ammonia. Finally, "moist combustion" was effected on 250 c.c. of the water, together with 500 c.c. of distilled water; the solutions employed and other details being in accord with Wanklyn's procedure. The estimations were made in triplicate. A small correction is made for the traces of organic matter in the half litre of distilled water. The results were in m.grms. per litre.

Free ammonia	0.188	0.194	0.192
Ammoniacal salts (NH_3)	0.359	0.364	0.362
Albumenoid	0.560	0.552	0.558
Organic matter in m.grms. of oxygen per litre	10.40	11.00	

There is every probability that the free ammonia found is far above the truth. The estimations were not made for some three months after the samples had been in the laboratory.

Composition of 1000 grms. of the general sample as compared with a published analysis of the English Channel.

	Sample.	Channel.*
Sodium chloride	28.023	27.059
Potassium chloride	0.770	0.766
Magnesium chloride	3.672	3.666
Magnesium sulphate	2.399	2.296
Calcium sulphate	1.365	1.406
Magnesium bromide	0.075	0.029
Calcium carbonate	0.129	0.033
	36.433	35.255
Water	963.567	964.745
	1000.000	1000.000

* Newth's "Inorganic Chemistry."

Comparison of Results between the present enquiry and the *Challenger* and Forchhammer figures (surface water) to 100 parts of halogen.

	Challenger.	Forchhammer.	Makin.
SO_3	11.576	11.88	11.908
Lime	3.053	2.93	3.154
Magnesia	11.212	11.03	11.721
Potash	2.405	1.93	2.410
Soda	74.760	Not determined.	73.664

Comparison of Results calculated to 100 parts of total salts.

	Challenger.	Makin.
Chlorine	55.292	55.185
Deduct O for Cl_2 and Br_2	12.493	12.454
	42.799	42.731
SO_3	6.410	6.593
CO_2	0.152	0.156
CaO	1.676	1.741
MgO	6.209	6.484
K_2O	1.332	1.334
Na_2O	41.234	40.783
Bromine	0.1884	0.1792
	100.0004	100.0012

By readjustment of the acid radicles with the oxides a further comparison may be made with the *Challenger* results, when the components are calculated to 100 parts of sea-water salts.

	Challenger.	Makin.
$NaCl$	57.758	76.915
$MgCl_2$	10.878	11.407
$MgSO_4$	4.323	4.483
$CaSO_4$	4.070	4.226
K_2SO_4	2.465	2.468
$MgBr_2$	0.217	0.206
$MgCO_3$	0.290	0.298
	100.001	100.003

This comparison shows perhaps more thoroughly the true variation between the waters of the North and South Atlantic and the mean results of the *Challenger's* investigations, extending as they did over nearly every quarter of the globe.

ON THE PREPARATION OF GLUCINUM BY ELECTROLYSIS.

By P. LEBEAU.

In the year 1828 Bussey and Wöhler isolated glucinum, almost simultaneously, by acting on its chloride with potassium. Since this time the separation of this metal has been the object of numerous trials, but in spite of all it remains even now a delicate laboratory experiment. We will recall specially Debray's researches on this subject. The very dissimilar physical and chemical properties attributed to glucinum by the different savants who have prepared it, have led us to try and find a process by which it can be obtained in a state always comparable with itself and of a constant purity.

The reaction generally employed in preparing glucinum consists in making an alkaline metal, potassium or sodium, react on the chloride of glucinum (Bussey, Wöhler, Debray, Nilson and Peterson, Reynold, Humpidge), or on the double fluoride of glucinum and potassium (Krusa and Morath).—*Liebig's Annalen der Chemie*, cclx., p. 187. A few attempts have been made to obtain the metal by means of electrolysis; MM. Nilson and Peterson (*Ann. de Chim. et de Phys.*, Series 5, xiv., p. 426) were not able to decompose the chloride, and found that its electrical resistance was so high that the current could not pass through it. M. Borchers (*Zeit. für Electrochemie*, 1895) claimed to have obtained glucinum by decomposing the triple chloride of glucinum, potassium, and ammonium, by a current of 1000 ampères per square metre of electrode and 5 volts, but he did not describe the metal obtained. Finally, Mr. Warren (*CHEM. NEWS*, lxxii., p. 310) claims to have made glucinum on a commercial scale, by electrolysing bromide of glucinum, by means of a current of 8 ampères and 12 volts. The metal obtained, converted into *objets d'art*, are at the present time in the possession of the Amir of Afghanistan. The bromide of glucinum used by Mr. Warren was apparently impure, for we have found that the melted halogen salts of glucinum do not conduct the electric current.

The glucinum compounds formed by chlorine, bromine, and iodine are rather difficult to obtain, and their rapid alteration in the presence of water causes them to be of very little practical use. We thought that perhaps the fluoride of glucinum, the properties of which are so little known, might present some advantages. We succeeded in preparing this body in a state of purity; it melts very easily, giving a fluid of great transparency, but unfortunately it is an absolute non-conductor of electricity. However, the addition of fluoride of sodium or potassium makes it a conductor, and then the glucinum can be separated by electrolysis.

Marignac (*Ann. de Chim. et de Phys.*, Series 4, xxx., p. 45) has studied the double fluorides, formed by fluoride of glucinum and the alkaline metals, from the crystallographical point of view, and he has obtained compounds of the forms $\text{GF}_2 \cdot 2\text{MF}$, and $\text{GF}_2 \cdot \text{MF}$. Notably the salts of sodium, $\text{GF}_2 \cdot 2\text{NaF}$ and $\text{GF} \cdot \text{NaF}$, which agree perfectly; the first melts at about 350° , giving, on cooling, a transparent vitreous mass; the second, which melts at a dull red heat, gives, on the contrary, a white mass with a crystalline fracture. This latter salt, having the lowest electrical resistance, would be chosen by preference when we have not a very strong current at our disposal.

We can prepare these salts by Marignac's method, which consists of mixing the concentrated solutions of the fluorides, and allowing them to crystallise; or we can

dissolve the pure hydrate of glucinum, and carbonate of sodium in the exact calculated proportions, in hydrofluoric acid. The liquid is evaporated to dryness, and the residue melted in a platinum crucible. This double salt may then be poured on to a sheet of platinum to cool, and kept in a stoppered glass bottle. If the quantity of fluoride of glucinum present is lower than that which corresponds to the formula $\text{GF}_2 \cdot \text{NaF}$ the product will become deliquescent.

The electrolysis is carried on very conveniently in a nickel crucible, which serves as the negative pole, the positive electrode consisting of a sheet or rod of graphitic carbon, which will not disintegrate under the influence of the current. We begin by melting the salt by means of a Bunsen burner; we then pass the current and withdraw the heat. The mass remains in fusion; too great a temperature is not needed, and it should not exceed a very dull red.

We had at our disposition the current produced by a small dynamo designed for charging accumulators, giving a normal current of 20 ampères and 80 volts. The current we used during the experiment was from 6 to 7 ampères, and from 35 to 40 volts.

After about 45 minutes, and using a crucible capable of containing 100 grms. of the salt $\text{GF}_2 \cdot 2\text{NaF}$, we obtained on the nickel crucible—above all in the median part—a metallic deposit formed of a non-adherent, felted, crystalline mass, which we isolated and treated with boiling water. After prolonged washings the disaggregation was complete, and we collected a powder formed entirely of rather irregular crystals, such as is often met with in electrolytic deposits, and which consists of pure glucinum entirely free from nickel and iron. Under the microscope it has a brilliant white metallic appearance; it does not contain any trace of amorphous matter.

We have, further, been able in these experiments to obtain alloys of glucinum, by working in carbon crucibles which served as the negative pole, and also contained the metal to form the alloy in a state of fusion: it is thus that we found it possible to prepare over again the glucinum bronzes, the preparation of which, by means of the electric furnace, we have previously described.—*Comptes Rendus*, cxxvi., No. 10.

NOTE ON THE SEPARATION AND ESTIMATION OF LEAD, COPPER, AND ARSENIC.

By F. JEAN.

This method consists in precipitating the hydrochloric solution, containing lead, copper, and arsenic, by a current of sulphuretted hydrogen, so as to separate these metals in the state of sulphides from the other bodies precipitable by sulphide of ammonium.

The precipitate of the mixed sulphides thus obtained is washed by decantation, then digested warm with a few drops of concentrated Javel water to oxidise the sulphides. When the solution is decolourised it is acidulated with a few drops of sulphuric acid and boiled to drive off the excess of chlorine. It is allowed to cool, and alcohol is added to render the sulphate of lead insoluble; filter, and from the weight of sulphate of lead found the percentage of lead present is calculated.

The filtrate, now free from lead, is warmed to drive off the alcohol; we then add ammonia in sufficient quantity to re-dissolve the precipitated arseniate of copper which forms at the commencement of the neutralisation.

The copper contained in this ammoniacal solution is titrated by means of a solution of sulphide of sodium. The final point of the titration is observed by placing one drop of an alkaline solution of lead on a white filter-paper by the side of a drop of the solution to be tested; the

moment the precipitation of the sulphide of copper is complete a brown arc is formed at the point where the two moist spots touch.

The titration of the copper being finished the solution is made acid by means of dilute hydrochloric acid, and the sulphide of copper is separated by filtration. It is necessary to acidulate before filtration, so as to be sure of separating a small quantity of sulphide of copper which is soluble in the alkaline solution. To the filtrate, containing arsenic acid, are added a few crystals of chlorate of potash; it is then concentrated by boiling to about 20 c.c.; we then nearly neutralise with ammonia, and add 5 c.c. of acetate of soda, and titrate the arsenic acid by means of an acid solution of acetate of uranium, observing the same conditions as when titrating phosphoric acid by Joulie's method.

The above method, applied to the analysis of the mixture, gave the following results:—

	Contained.	Found.
Lead	0.122 grm.	0.1212 grm.
Copper	0.020 "	0.020 "
Arsenic	0.0375 "	0.0370 "

—*Journ. de Pharm. et de Chimie*, vii., No. 5.

A SIMPLE AND ACCURATE METHOD OF TESTING DIASTATIC SUBSTANCES.

By JOKICHI TAKAMINE.

THERE are various methods known for determining the diastatic power of substances, as Lintner's, Junk's, and others. While some of these are very reliable in many respects they are complicated, and require specially trained hands to get reliable results. They are not, therefore, applicable when quick, simple, and accurate testing is desired, as in diagnosing a certain form of amyloitic dyspepsia by determining the diastatic power of the patient's saliva. My proposed test method is based upon the stable diastatic property of Taka-diastase.

So far as is generally known, and it is also my own experience, the diastase isolated from malt, and that precipitated principle of saliva known as ptyaline, lose their diastatic power by standing. Therefore, when the diastatic power of any substance is to be determined, it is necessary, if they are used, to go through the long process each time of determining the quantity of sugars formed by their action on starch (Lintner's method), or else measure the length of time required to convert the given quantity of starch into sugar (Junk's method). While these processes have valuable merits of their own, yet they have the disadvantage of being rather complicated for quick every-day work. In carrying out my proposed method, a quantity of Taka-diastase is tested by either of the above-mentioned processes (Lintner's or Junk's*), and its exact diastatic power determined once for all. The diastatic power may be expressed as, say, 300 Lintner's units, or it may be expressed as converting 100 times its own weight of dry starch into sugar in ten minutes.

The diastatic power of any substance under examination is now compared with the standardised sample, and expressed in any terms desired, either directly, or by simple calculation. It is highly desirable that one standard should be adopted, and, whatever that may be, the following comparative test will be found useful. First prepare the following solutions:—

1. *Standard Taka-diastase Solution*.—Dissolve 1 grm. of standardised Taka-diastase solution in 100 c.c. of water; this solution ought to be made fresh each day.

2. *Starch Solution*.—Make a 5 per cent solution of

neutral potato starch by boiling 800 c.c. of distilled water in a suitable wide mouth vessel; pour into it milk of starch, made by stirring 50 grms. of starch into 200 c.c. of cold water, and boil two minutes.

3. *Iodine Solution*.—Place 1 grm. of iodine and 2 grms. of potassium iodide in a flask, add a little water, say 5 c.c., agitate until dissolved, and dilute to 120 c.c.; or dilute 50 c.c. tincture of iodine, U.S.P., with 50 c.c. of water containing 2.5 grms. of potassium iodide.

Apparatus Required.—(1) One quart agate-ware kettle; (2) one shallow tin pan, 2 inches deep, 8 inches in diameter; (3) two 1 c.c. pipettes graduated to tenths; (4) eight large glasses or tumblers of about 150 c.c. capacity each; (5) ten small test-tubes; (6) one 100 c.c. cylinder; (7) two white dinner plates.

Process of Testing.—Pour into each of the eight glasses 100 c.c. of the hot starch paste. Place them side by side in the shallow pan of warm water at about 40° C. Measure into the first glass 1 c.c. of the saliva or other liquid to be tested. Pour, of the standard diastase solution, in quick succession—

Into the second glass		1 c.c.
" third "		2 "
" fourth "		3 "
" fifth "		4 "
" sixth "		5 "
" seventh "		6 "
" eighth "		7 "

Then the contents of each glass is stirred with the test-tube as a stirring rod in quick succession, until the starch paste all becomes limpid. At this stage it will be observed that the stronger the diastatic power the quicker the liquefaction of the paste. When the contents of the glasses become liquefied, take out of each glass in succession a drop of the liquid by means of the stirring test-tube, and drop on a white dry dinner plate in the order of the glasses. When there are eight drops of equal size on the plate, drop on each one drop of the iodine solution. Then spread each sample with the finger to about the size of a silver dollar. The drops from the second to the eighth glass will form a colorimetric scale from blue to purple and reddish brown. Observe now which member of the scale corresponds to the colour of the one containing the saliva. The comparison is made more certain by repeating the tests within the first ten minutes after the saliva is put in.

Suppose the colour corresponds to somewhere between the fourth and fifth, then we can assume it at 4.5, and calculate the diastatic strength in terms of starch converted or sugar formed; or, if further accuracy of the test is desired, a scale of starch glasses containing standard diastase solution of 4 c.c., 4.2 c.c., 4.4 c.c., 4.6 c.c., 4.8 c.c., and 5 c.c., may be put up and compared with 1 c.c. of the given saliva in the same manner.

Instead of having only one specimen at a time, several samples of saliva or other diastatic substances can be tested at once.—*American Journal of Pharmacy*, vol. lxx., No. 3.

THE DIGNITY OF ANALYTICAL WORK.*

By C. B. DUDLEY.

It will doubtless be conceded by all, that in the choice of the field to which one proposes to devote his life-work, a number of things should be consulted. Among these may be mentioned not only mental capacity and the opportunities for training by courses of study, which may be available to him, but also what may be termed natural inclination or love for the work. Just how much weight should be given to each of these elements is a query not easily answered, but few will deny that genuine interest

* Lintner, *Journ. für Prakt. Chem.*, [2], xxxiv., 378–394. Junk, *Am. Journ. Pharm.*, lv., 289, and lvii., 13; modified in *Bulletin of Pharmacy*, February, 1898, p. 52.

* Presidential Address delivered at the Washington Meeting of the American Chemical Society, December 29, 1897. From the *Journal of the American Chemical Society*, vol. xx., No. 2.

in or real love for the field of work chosen, should be allowed as great sway as possible. Those of us who have gotten far enough along in our life-work to be able to look back somewhat, and to see and differentiate the causes that have shaped our line of effort, know full well that circumstances beyond our control, rather than our inclinations and desires, have in many cases determined our course, but the fact nevertheless remains, that for the best results, for the attainment of even moderate success, one's efforts must be in a field agreeable to him, and his heart must be in his work. Fortunate is the man for whom circumstances so shape themselves, that he is able to pass his years in the field of his choice, and spend and be spent in work that is congenial to him.

Assuming now that for most of us, circumstances and conditions have been such that we are spending our lives in the field of our choice, let us consider for a moment a tendency that seems to be a concomitant of those thus fortunately situated. Do we not occasionally find in ourselves a disposition to magnify the importance of the field in which we happen to be engaged? Are we not somewhat inclined, quite naturally perhaps, to think that our field of work is more important than that in which others are occupied? Does not the theoretical chemist, whose inclinations lead him to spend his time in writing reactions, and building structural formulæ of wondrous architecture, often feel within himself that his work is on a higher and nobler plane than that of the patient analyst, who has furnished the data which he uses? Does not the organic chemist who delights in the study of the carbon compounds, who can repeat for you series after series of chemical bodies, differing from each other by the constant addition of an element or group of elements, in whose vocabulary "types," "substitutions," "replacements," "condensations," and "isomers" are familiar words, and who, when a new organic compound is discovered, cannot rest until he has found to what series, and what place in the series, it belongs, or what its relations are to other bodies in that marvellous structure, based on the element carbon, which the studies of the last half century have reared before our eyes, I say does not this organic chemist oftentimes feel that he is engaged in a field far more worthy of study, and to which is due much more consideration than to that of his inorganic brother, who devotes days and perhaps weeks to unraveling the constitution of some obstinate silicate, whose crystalline form gives little help, and whose oxygen ratio is hidden or obscure? Or again, does not the physical chemist oftentimes think, that with the tools of his more especial field, with his specific heats, his vapour-densities, his heats of chemical combination, and his ions, he is quite competent to solve all problems worth solving in the realm of chemistry, and that those who are engaged in other lines are far below his standard, and can be looked down upon quite with pitying sympathy? Still once more, do we not often see the pure chemist whose battle cry is "original work for the work's own sake," claim for himself the highest seat in the synagogue, and refuse to join his efforts with those of others whom he regards as his humbler brethren, viz., those working in the field of applied chemistry, in securing the benefits of organisation to extend and widen the borders of our science? Finally, not to make distinctions, do we not frequently see the analyst who knows so well how necessary it is to have the trained and skilful hand, and the acute and watchful brain, both working together, and at the same time, in order to secure the accuracy, without which his work is worthless, claiming for his field, that it is the foundation upon which our science rests, and that those who spend their time in locating the position of an atom in its molecule, or in finding the relations of an organic compound to other members of its series, or perchance in inventing long names for new compounds in which all the resources of the ancient Greek and Latin are brought to bear, to reveal in one word the constitution of the compound, I say does not the analytical chemist

often regard these workers as shallow, empty headed, and unworthy to be called chemists.

Now far be it from me to say that this partiality of each for his own field is blameworthy. We can, indeed, conceive of cases in which this partiality may be carried a little too far, but within proper limits, not only is it not blameworthy, but even as it seems to us, it may be praiseworthy for one to magnify the importance of the work in which he is engaged. A just and proper estimate of the value of his own work, a reasonable pride in his chosen science, or in that paddock of his science which it has fallen to the share of each to care for and cultivate, and indeed a moderate, though necessarily a somewhat partial, comparison of himself and his field of labour, with others, even though that comparison is somewhat to the detriment of the others, are not always necessarily bad. On the other hand, such pride and such comparisons tend to stimulate to renewed activity, tend to sustain in the perplexities and discouragements of work, and tend to keep one's effort concentrated on the work which he can do best. Looked at in this light, the generous rivalry of one branch of our science with another, or the pardonable pride of each in his own chosen field, and even in his own work, may be a distinct advantage, and I know you will bear with me a few minutes, while I, with proper modesty, and in the true spirit, I hope, try to magnify a little the field of analytical work.

To my mind, then, it is just and proper to take pride in analytical chemistry, because of the power which a properly conceived and executed analysis has of explaining difficulties. A few illustrations will, perhaps, make this point clear, and I am sure I will be pardoned for giving illustrations from my own experience, rather than historical ones.

Some years ago, after a passenger coach on the Pennsylvania Railroad had been through the hands of the car cleaners, it was noticed by some of the officers that the paint on the outside looked very badly, and had apparently been injured by the cleaning. A careful examination by the paint experts revealed the fact that the varnish was nearly all gone, and in some places the paint itself partially removed. As a matter of discipline, the car cleaners were called to account, and requested to explain why the paint and varnish had been so badly injured. Their reply was that with the soap that was furnished for car cleaning no better results could be obtained. This statement was, of course, received with a grain of allowance, it being well known to railroad operating officers, that almost universally when anything goes wrong, and the men are called to an account, the materials are blamed. However, in order to give the men the benefit of the doubt, a sample of the soap was obtained and submitted to analysis, when it was found that this soap actually contained over 3 per cent of free caustic soda and about 7 per cent of sodium carbonate. It is evident that this soap had been very carelessly made from cheap materials, and since it is well known that water solutions of both caustic and carbonated alkalies are fairly good solvents for dried linseed oil and other constituents of paint and varnish, it is clear that the defence of the men, in this case at least, was legitimate, and that the soap was really at fault. It may be added for information, that the circumstances above described led to the preparation of a specification for common soap, in which the amount of free and carbonated alkali was limited to very low figures, and that no similar difficulty of destruction of paint and varnish has since occurred.

(To be continued.)

Osmotic Pressure and Cryoscopy.—A. Reyhler.—This is a long mathematical paper in which the author criticises Mr. H. Crompton's deductions, that the law of van't Hoff requires a correction, and he shows that the facts given by Mr. Crompton agree very well with van't Hoff's law without any modification whatever.—*Bull. Soc. Chim. de Paris*, Series 3, vol. xix.-xx., No. 3.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Anniversary Meeting, March 31st, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

MR. W. G. McMILLAN and Dr. THORNE were appointed scrutators, and a ballot was opened for the election of Officers and Council for the ensuing year, the ballot being closed at the conclusion of the President's Address.

The PRESIDENT, in beginning his Address, remarked that the papers submitted to the Society during the past session included original work in all branches of chemical science. They have been contributed, not only from the laboratories of the older universities of Oxford and Cambridge, but also from the university colleges and other institutions throughout the country. The publications of the Society are, therefore, a complete record of English chemical Science, and of the researches pursued at different educational centres throughout the country.

The *Proceedings* are now the medium of publication, not only of abstracts of papers which will subsequently appear in the *Transactions*, but also of the rapid publication of short papers and preliminary notices.

The rule, which came into force last year, requiring that authors shall deposit their papers and abstracts with the Secretaries before they are officially announced for reading at a meeting, has worked well; it has made it possible to expedite the publication of the *Proceedings*, and to send advance proofs to the authors for correction.

The meetings have been well attended, the meeting-room being often quite full. Occasionally, interesting discussions have taken place, but as so many of our papers deal with details and matters of fact, anything like a debate must necessarily be of rare occurrence.

The following Past-Presidents, Lord Playfair, Dr. A. W. Williamson, Sir E. Frankland, Dr. W. Odling, Sir F. A. Abel, Dr. J. H. Gladstone, Sir J. H. Gilbert, this year complete a connection of fifty years with the Society. To mark its sense of the great services they have rendered to chemical science, the Council have resolved to entertain them, in the name of the Society, at a dinner on June 9th, given to commemorate their half century of Fellowship of the Society.

During the past year the Council has had under consideration the question of a revision of the Bye-laws. A Committee was appointed early in the year, which has met several times during the session, but, in view of the wider questions which have occupied the attention of the Council, they have not been able to complete their report.

During the session a section of the Fellows residing at a distance from London have expressed a desire to take part in the election of Officers and Council without being forced to undertake a journey to London to register their votes at the Annual General Meeting. A Memorial was drawn up, asking the Council to prepare and lay before the Society a Bye-law enabling Fellows to record their votes by post. This Memorial was signed by 540 Fellows, of whom 400 resided outside the London district. In the meantime, the Bye-law Committee had taken steps to ascertain the powers of the Society in this matter under their Charter. They were legally advised that any Bye-law framed to admit voting by post or proxy would be repugnant to the Charter, and therefore invalid.

When the Memorial was presented to the Council, a covering letter contained another suggestion, viz., that it might probably be necessary to obtain a Supplemental Charter, in order to enable the Council to carry out the wishes of the Memorialists. This suggestion was seriously considered by the Council, and further legal opinion was taken on the subject. They were advised that no application for a Supplemental Charter which was not supported by the practically unanimous wish of

the Fellows would be listened to by the Privy Council. They have since been further advised that it would be *ultra vires* on their part to expend any part of the funds of the Society in applying for a Supplemental Charter. All the documents relating to the Memorial and the various legal opinions, together with the decisions of the Council, have already appeared in the *Proc. Chem. Soc.*, 1898, pp. 1, 33, 61.

The following eminent foreign chemists, whose names are familiar to us all as of those who have advanced knowledge in various departments of our science, were elected Foreign Members in January of this year, bringing the total number of Foreign Members of the Society up to 38:—Professors S. Arrhenius, Th. Curtius, A. P. N. Franchimont, W. Körner, W. Markownikoff, N. A. Menshutkin, H. Moissan, W. Ostwald, F. M. Raoult, I. Remsen, W. Spring, L. J. Troost, P. Waage, J. D. van der Waals. The Society has lost a Foreign Member by death: Professor Victor Meyer, one of the most brilliant and original of the modern school of chemists. The Kekulé Memorial Lecture has been delivered by Professor Japp. It has been resolved that the Memorial Lectures delivered up to 1896 shall be bound and published as Vol. I. of the Society's Memorial Lectures as soon as possible.

The Council have nominated the Treasurer and Editor as Delegates for the Society to the National Committee appointed by the Royal Society to prepare a Catalogue of Scientific Literature.

The remainder of the President's Address was devoted to an account of recent advances in Low Temperature Research, which will subsequently appear in the *Transactions*.

The numerical strength of the Society was as follows:—

Number of Fellows, March 31st, 1897	2079
since elected	114
reinstated by Council	4
	2197

Removed on account of non-payment of two annual subscriptions	17
Withdrawn	22
Deaths	18
	57

Number of Fellows, March 31st, 1898	2140
Foreign Members	38

Fourteen Foreign Members were elected during the year.

The following have died:—J. J. Bowrey, G. W. Child, E. H. Gaskell, B. H. Gibbins, W. A. L. Hammersley, S. J. Harris; Walter Jardine, G. A. Keyworth, M. H. Lackersteen, Samuel Lees, Herman Lescher, Thomas Mitchell, Howard Newton, F. M. Rimmington, J. W. Rodger, Taraprasanna Roy, W. J. Saint, James Napier.

The number of communications made to the Society during the year was 127.

One hundred and fourteen papers were published in the *Transactions* for 1897, occupying 1204 pages, whereas in the preceding year 117 papers were published, occupying 1702 pages.

The following were the statistics relating to the Abstracts. (See next column).

Eight hundred and fifty volumes had been borrowed from the Library. The additions comprised 78 books, 282 volumes of periodicals, and 36 pamphlets.

Sir W. CROOKES, F.R.S., proposed a vote of thanks to the President, coupled with the request that he would allow his Address to be printed in the *Transactions*.

Dr. W. J. RUSSELL, F.R.S., seconded the motion, which was carried by acclamation.

The PRESIDENT having returned thanks, Dr. THORPE, F.R.S., the treasurer, gave an account of

PART I.		
	Pages.	No. of Abstracts.
Organic Chemistry	648	1049
PART II.		
General and Physical Chemistry		324
Inorganic Chemistry		270
Mineralogical Chemistry		192
Physiological Chemistry		168
Chemistry of Vegetable Physio- logy and Agriculture		158
Analytical Chemistry		414
Total in Parts I. and II.	612	1526
	1260	2575

the balance sheet, which he laid before the Society, duly audited.

The receipts had been:—By admission fees and subscriptions, £3827; by sale of Journal and advertisements, £686 3s. 6d.; and by dividends on invested capital, £414 6s. 0d. The expenses had been:—On account of the Journal, £2963 11s. 1d.; on account of the Proceedings, £278 7s. 10d.; on account of the General Index, £182 18s. 1d.; on account of the Library, £307 4s. 9d.; House expenses, £203 6s. 7d.; the total expenditure being £4807 6s. 8d. Grants amounting to £150 had been made to Fellows from the Research Fund during the year.

Dr. GLADSTONE, F.R.S., proposed that the thanks of the Fellows be tendered to the Treasurer for his services during the past year; this motion was seconded by Dr. STEVENSON, and carried.

The TREASURER, in responding, proposed a vote of thanks to the auditors.

Dr. ATKINSON seconded the motion, which was unanimously adopted, and acknowledged by Mr. R. J. FRISWELL.

Mr. D. HOWARD proposed a vote of thanks to the Officers and Council.

Mr. CASSAL seconded the motion, which was unanimously adopted.

Prof. THOMSON, F.R.S., responded on behalf of the Council.

Dr. B. DYER proposed a vote of thanks to the Editor, Sub-Editor, Abstractors, and Indexers, which was seconded by Mr. R. J. FRISWELL, and carried.

Mr. GROVES, F.R.S., responded.

The scrutators having presented their report to the President, he declared that the following had been duly elected:—

President—James Dewar, M.A., LL.D., F.R.S.

Vice-Presidents who have filled the office of President—Sir F. A. Abel, Bart., K.C.B., D.C.L., F.R.S.; H. E. Armstrong, Ph.D., LL.D., F.R.S.; A. Crum Brown, D.Sc., LL.D., F.R.S.; Sir W. Crookes, F.R.S.; Sir E. Frankland, K.C.B., D.C.L., F.R.S.; Sir J. H. Gilbert, Ph.D., LL.D., F.R.S.; J. H. Gladstone, Ph.D., D.Sc., F.R.S.; A. Vernon Harcourt, M.A., D.C.L., F.R.S.; H. Müller, Ph.D., LL.D., F.R.S.; W. Odling, M.B., F.R.S.; W. H. Perkin, LL.D., Ph.D., F.R.S.; Lord Playfair, G.C.B., LL.D., F.R.S.; Sir H. E. Roscoe, LL.D., F.R.S.; W. J. Russell, Ph.D., F.R.S.; A. W. Williamson, LL.D., F.R.S.

Vice-Presidents—F. R. Japp, M.A., LL.D., F.R.S.; G. D. Liveing, M.A., D.Sc., F.R.S.; William Ramsay, Ph.D., LL.D., F.R.S.; J. Emerson Reynolds, M.D., D.Sc., F.R.S.; John M. Thomson, F.R.S.; William A. Tilden, D.Sc., F.R.S.

Secretaries—Wyndham R. Dunstan, M.A., F.R.S.; W. P. Wynne, D.Sc., F.R.S.

Foreign Secretary—Raphael Meldola, F.R.S.

Treasurer—T. E. Thorpe, LL.D., F.R.S.

Other Members of Council—P. Phillips Bedson, D.Sc.; E. J. Bevan; H. J. H. Fenton, M.A.; W. Gowland;

Otto Hehner; C. T. Heycock, M.A., F.R.S.; D. Howard; Herbert McLeod, F.R.S.; Rudolph Messel, Ph.D.; H. Forster Morley, M.A., D.Sc.; Alexander Scott, M.A., D.Sc.; Arthur Smithells, B.Sc.

List of Fellows.

A new list of Officers and Fellows of the Chemical Society being in course of preparation, it is requested that Fellows will send any alteration of address, without delay, to the Assistant Secretary, Burlington House, London, W.

NOTICES OF BOOKS.

The Calorific Power of Fuels, founded on Scheurer-Kestner's "Pouvoir Calorifique des Combustibles." With the addition of a very full Collection of Tables of Heats of Combustion of Fuels, Solid, Liquid, and Gaseous. To which also is appended the Report of the Committee on Boiler-tests of the American Society of Mechanical Engineers (December, 1897); Tables of Constants used. By HERMAN POOLE, F.C.S., Member of [several Learned Societies], First Edition, First Thousand. New York: John Wiley and Sons, London: Chapman and Hall, Ltd. 1898. Pp. xv.—255. 8vo. Illustrated.

THE author first planned a mere translation of Scheurer-Kestner's "Pouvoir Calorifique des Combustibles," but he found it necessary to make so many changes to adapt it to American methods and data that he retained only the skeleton of the French treatise, and filled this in from other sources. In the Introduction he adopts the usual classification and definition of fuels, and then explains the difference between calorific power and heat of combustion. He discusses the method of determining the heat of combustion, and then treats the subject of calorimetry quite exhaustively. The divers forms of calorimeters and the manner of using them occupy four chapters; following these are chapters on solid, liquid, and gaseous fuels. Chapter X. deals with the calorific power of coal burnt under a steam boiler; Chapter XI. treats of air supplied and gaseous products of combustion; and Chapter XII. explains the method of calculating the heat units.

In the Appendix is the Report named on the title-page and fifteen tables of constants. Following these are fuel tables, giving analyses of a large number of fuels of the three classes, together with their heat units and the authorities. The coals in these tables are from the chief beds in the United States, as well as in Nova Scotia, Ontario, Chili, France, Great Britain, Austria, Hungary, Germany, Spain, Russia, and New Zealand. Lignites and oils from America and abroad are treated in similar tables. An Index closes a well-printed volume, which is quite free from typographical errors, though "fused" (page 176) is an unusual way of spelling fused.

The work will undoubtedly prove of service to those engaged in the examination of fuels and the determination of their economic value.

Quantitative Chemical Analysis by Electrolysis. By ALEXANDER CLASSEN in co-operation with WALTER LÖB. Authorised Translation. Third English, from the Revised and greatly enlarged Fourth German Edition, by WILLIAM HALE HERRICK and BERTRAM B. BOLTWOOD. New York: John Wiley and Sons, London: Chapman and Hall, Ltd. 1898. Pp. xii.—301. 8vo.

This standard treatise, by the Professor of Electro-Chemistry in the Royal School of Technology at Aachen, revised by the Lecturer on the same subject, differs from the earlier editions by the insertion of a Section devoted

to the ion theory. Faraday's law, Ohm's law, the significance of tension, of current strength and of resistance, as well as the theory of electrolytic precipitation, are all clearly explained. The various forms of voltmeters, galvanometers, and the ampèremeter for measuring the current strength, and the voltmeter and electrometer for measuring tension, are well described and illustrated. Under the caption "sources of current" the authors describe the divers forms of cells, storage batteries, electro-magnetic machines, and thermo-electric piles.

A very practical chapter (not numbered) gives instructions for carrying on the process of analysis, with numerous woodcuts showing disposition of the apparatus: the translators have here inserted an account of von Malapert's apparatus.

A brief historical sketch of the development of electrolysis is rather singularly placed, being between chapters on the process of analysis and arrangements for analysis; credit is given to F. W. Clarke, Edgar F. Smith, and W. Gibbs, for their improvements and discoveries.

The former and the present equipment of the Electrochemical Institute of the Technical High School at Aachen are explained in detail, and illustrated with three full-page reproductions of excellent photographs. In Section II., also called "Special Part," the quantitative methods of determining metals and of their separation are described; at the beginning of each Section twelve to twenty references to literature are given—a valuable feature for those desirous of consulting original papers. In the Appendix are supplied examples for instruction. The book closes with two indexes.

The authors, translators, and publishers deserve credit for jointly producing so valuable and handsome a volume.

H. C. B.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xix.-xx., No. 3.

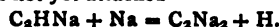
An Apparatus for Facilitating the Separation of the Natural Organic Principles.—C. Chabré.—This paper cannot be understood without the accompanying diagram.

A Double Carbonate of Soda and Protoxide of Chromium.—G. Bauge.—If to a well-washed and still damp chromous acetate we add a solution of carbonate of soda in boiled water, we see that the acetate immediately goes into solution, and that almost immediately a reddish brown body is precipitated. This precipitation must be carried on in an atmosphere of carbonic acid perfectly free from oxygen. This sodic chromous carbonate forms two hydrates, one containing 10 molecules of water and the other 1 molecule. The former is a reddish brown powder, showing lozenge-shaped crystals under the microscope; it is an energetic reducing agent; it decomposes water below 100°, giving off hydrogen. In vacuo it loses water at the ordinary temperature; at 100° it forms the 1 molecule hydrate. Dilute sulphuric and hydrochloric acids dissolve it, giving blue solutions. The salt with 1 molecule of water is a yellow powder, with very similar properties to the other one; heated in vacuo or in a current of hydrogen it changes its colour to a dark brown, becoming yellow again on cooling. Cold boiled water gradually changes it into the salt with 10 molecules of water; when heated in a current of chlorine it gives chloride of chromyl. Analyses give these two compounds the following formulae:—



Decomposition of Chloroform, Bromoform, and Chloral by an Aqueous Solution of Potash.—A. Desgrez.—Not suitable for abstraction.

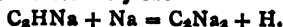
Preparation of Carbide of Sodium and Monosodic Acetylene.—C. Matignon.—To prepare monosodic acetylene a current of dry acetylene is drawn through a flask containing sodium; from thence first into an empty flask and then into a large flask containing glycerin; the flask containing the sodium is placed on an oil-bath, and as soon as the sodium begins to melt the flask is shaken; the reaction then proceeds regularly, still shaking the flask and plunging it into the oil from time to time to keep up the temperature, which should be 180°. The sodium becomes gradually pulverised, and the reaction is at an end when it appears as a white homogeneous powder. To prepare carbide of sodium the same apparatus is used, but the operation is much quicker; the temperature is rapidly brought up to 220°, and kept at from 220—230°; the monosodic carbide which forms below 200° is then decomposed by the sodium not yet attacked—



Action of Sodium on Acetylene.—C. Matignon.—Between its melting-point and 190° sodium decomposes acetylene, $\text{C}_2\text{H}_2 + 2\text{Na} = \text{C}_2\text{NaH} + \text{H}$. Above 210° both the atoms of hydrogen are substituted and carbide of sodium is formed, $\text{C}_2\text{H}_2 + \text{Na}_2 = \text{C}_2\text{Na}_2 + \text{H}_2$. From 210—220° monosodic acetylene is decomposed—



At the same temperature sodium transforms monosodic acetylene into disodic acetylene—



All these reactions take place quantitatively, producing pure, perfectly white bodies. At 300° acetylene no longer acts on a 2 per cent sodium amalgam.

Contribution to the Study of the Sulphonic Derivatives of Camphor.—A. Reychler.—In this paper the author describes at great length the only practical way of effecting the sulphonation of camphor. He has principally studied crystallised camphosulphonic acid and described some of its most important derivatives. By its oxime it would probably give rise to a sulphonic camphonic acid. Under the influence of reducing agents it should give interesting thio-derivatives.

MEETINGS FOR THE WEEK.

MONDAY, 18th.—Society of Arts, 8. (Cantor Lectures). "Sources of Commercial Indiarubber," by Dr. D. Morris, C.M.G.

TUESDAY, 19th.—Royal Institution, 3. "Phases of Art—Past and Present," by T. C. Goch.

WEDNESDAY, 20th.—Society of Arts, 8. "Stage Mechanism," by Edwin O. Sachs.

— Microscopical, 8. "On some Organic Substances of High Refractivity Available for Mounting Specimens for Examination under the Microscope," by H. G. Madan, M.A., F.C.S. "Instantaneous Photomicrography," by E. B. Stringer, B.A. At 7.30, an Exhibition of Diatoms, by H. J. Morland.

THURSDAY, 21st.—Chemical, 8. Ballot for the Election of Fellows. "The Carbohydrates of Barley Straw," by G. F. Cross, E.J. Bevan, and Claude Smith. "Isomeric Boroniamines," by M. O. Forster, Ph.D.

— "Some Derivatives of Benzophenone," by F. E. Matthews, Ph.D. "Researches on Camphoric Acid," by S. B. Schryver, Ph.D.

— Royal Institution, 3. "Some Leaders in the Poetic Revival of 1760-1820—Cowper, Burns, and Scott," by The Rev. Canon Alinger, M.A., LL.D.

— Society of Arts, 4.30. "Recent Railway Policy in India," by Horace Bell, M.Inst.C.E.

FRIDAY, 22nd.—Royal Institution, 9. "The Recent Eclipse," by W. H. M. Christie, C.B., M.A., F.R.S., Astronomer Royal.

— Physical, 5. "On a Method of Viewing Newton's Rings," by the Rev. T. C. Porter.

SATURDAY, 23rd.—Royal Institution, 3. "Programme Music" (with Musical Illustrations), by Sir Walter Parratt, Mus. Doc., Master of the Queen's Music.

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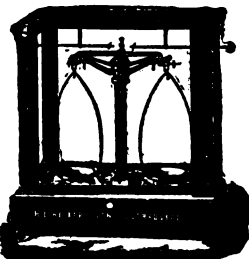
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Fig. 11. No. 91.

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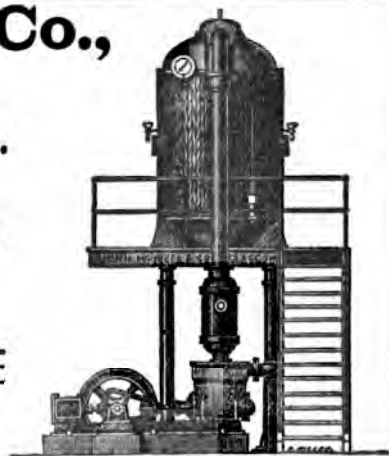
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CONTENTS.

ARTICLES:—	PAGE
Separations from Chromic Acid, by Harry Brearley.....	179
On Nascent Hydrogen, by D. Tommasi.....	180
On a Simple Method of Preparing Hydronitric Acid, by M. Dennstedt and W. Göblich.....	181
London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.....	183
On the Ammonia Test of Cocainum Hydrochloricum according to MacLagan, by Messrs. C. F. Boehringer and Soehne.....	183
The Dignity of Analytical Work, by C. E. Dudley.....	185
CORRESPONDENCE.—Volumetric Determination of Chromium in Iron and Steel.....	187
NOTICES OF BOOKS.—An Elementary Treatise on the Metric System of Weights and Measures—Sugar, Home-grown and Home-manufactured.....	187
CHEMICAL NOTICES FROM FOREIGN SOURCES.....	188
MEETINGS FOR THE WEEK.....	190

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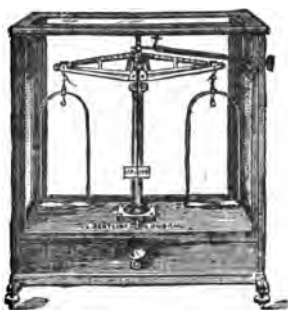
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FIG. 3A.

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SEPARATIONS FROM CHROMIC ACID.

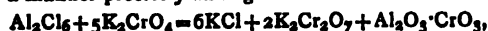
III. THE SEPARATION OF ALUMINUM.

By HARRY BREARLEY.

Reaction between K_2CrO_4 and Al_2Cl_6 .

In a preceding paper (CHEM. NEWS, lxxvii., p. 49) it is stated, on the authority of Storer, that until five equivalents of potassium chromate have been added to a solution of ferric chloride, any precipitate formed locally is redissolved on shaking. This statement, using a solution of bar iron in aqua regia, I have not been able to verify. A precipitate forms much earlier than it should, and indeed can be made to form with variable amounts of chromate by suitably prolonging the addition of this reagent. The precipitates form much earlier in hot than in cold solutions.

These facts are in sharp contrast to the behaviour of aluminum (used as chloride). The reaction is formulated in a manner precisely analogous to that of iron—



and Storer and Eliot state similarly that no permanent precipitate is formed until five equivalents of the chromate have been added. This statement has been confirmed under just such conditions as with iron compounds would most emphatically refute it, viz., with a hot solution. In a cold solution the reaction proceeds very unsatisfactorily. Thus the precipitate formed almost from the first dissolves very tardily and never completely, except with very prolonged standing. If a slight opalescence be disregarded, and further addition of the chromate made, it can readily be seen that the precipitates are partly dissolved, but the turbidity which is apparently permanent continually increases, and is hardly effected by standing two or three hours, although only half the theoretical amount of chromate has been added.

Only so many of the properties of these basic chromates as are necessary to explain the respective separations need be noticed here; it has become evident that the strict analogy between them, insisted upon by Storer and Eliot, should be further examined.

Reaction between K_2CrO_4 and $Al_2(HO)_6$.

The aluminum hydrate ($=\frac{1}{2}$ gm. Al) was prepared by adding an equivalent quantity of alkali to an acid solution of the chloride; in I. ammonium carbonate and in II. soda carbonate was used for this purpose, the chromate being added immediately after the alkali, and then the whole raised to boiling. There was recovered from the filtrates the amounts shown in—

TABLE VI.

K_2CrO_4 added.	Found in filtrate.	
	I.	II.
10 c.c.	3.2	4.5
30 "	14.1	18.2
50 "	—	33.9

Relation of these Reactions to Analytical Methods.

Most of the methods previously noticed as being applied to the separation of iron are said to be also applicable to the separation of aluminum from chromic acid. A trial of several in turn was made; the results are collected into the following tables.

Separation with Ammonium Hydrate, Ammonium Carbonate, and Soda Carbonate.

In every case the alkali was two normal strength; weight of Al, half a gm.; volume of K_2CrO_4 (1 c.c. = 0.05 Fe), 50 c.c. The arrangement of the results explain the details of the operations.

TABLE VII.

Excess of alkali.	Percentage separation.		
	$(NH_4)HO$.	$(NH_4)_2CO_3$.	Na_2CO_3 .
10 c.c.	89.0	81.3	94.0
20 "	90.5	93.6	95.3
30 "	93.1	94.4	99.0

The figures would seem to indicate the direction in which a perfect separation might be sought, but according to common knowledge the larger excesses of alkali would dissolve appreciable quantities of the precipitate $Al_2(HO)_6$. This defect is more marked with the soda than with the ammonia carbonate. One may therefore choose between leaving chromic acid in the precipitate or losing aluminum hydrate in the filtrate. Where the subsequent estimation of the elements is a gravimetric one the errors tend to balance each other, and for this reason may have seemed of less moment than they actually are.

Fresenius ("Quantitative Analysis," p. 294), after separating with ammonia or its carbonate, says, "If the washed alumina has a yellow colour, treat it on the filter with ammonia, and wash with boiling water; this will remove the last traces of chromic acid. A little alumina, however, dissolves in the ammonia, therefore heat the ammoniacal fluid in a dish until it has almost lost its alkaline reaction, and filter the separated flocks of alumina to the principal precipitate." It has been stated (CHEM. NEWS, vi., 182) that the basic chromates of alumina, when thoroughly washed, leave pure hydrate of alumina on the filter, but an acid chromate of alumina passes into the filtrate. If this is so, it is necessary *always* to examine the filtrate of an alkaline separation of aluminum and chromic acid. In any case the means proposed by Fresenius for recovering the dissolved alumina are open to the objection illustrated by Table VI., and the retention of this recovered alumina on the previously used filter tends to very considerably increase this error. On page 232 of the last edition of "Select Methods" one is instructed to separate alumina from chromic acid by adding ammonia, and boiling until the smell becomes very faint. The method is part of an electrolytic separation after Classen.

Separation with Soda Acetate.

Solutions of aluminum chloride, like solutions of ferric chloride, may be "neutralised,"—that is, they are able to re-dissolve their freshly precipitated hydrate. The extent to which this may take place has been carefully examined, and will be stated later; all we need remark now is, that under the most favourable conditions the—



is more basic than the similar iron compound. From this it follows, if the solution be neutralised, that an acetate separation becomes subject to error, first, on account of the chromate's reaction with the chloride, and, second, on account of its reaction with the hydrate, which becomes precipitated as the chloride is decomposed.

Tests were made on solutions both with and without "neutralising." The filtrate from the samples not "neutralised" contained large quantities of alumina. This may be mostly due to the fact that the solutions were cooled before filtering (see Table I.), though partly it might be accounted for by the known solvent action of soda acetate, and the possible presence of acetic acid due to the decomposition of the precipitated aluminic acetate. The acetate contained 500 grms. of the crystallised salt per litre.

TABLE VIII.

Soda acetate.	Percentage separation.	
	"Neutralised."	Not "neutralised."
20 c.c.	77.2	—
30 "	82.4	—
50 "	84.4	79.2
100 "	—	84.6
150 "	—	85.0

In each case the separation was of half a gram. of Al from 50 c.c. of the K_2CrO_4 solution in a bulk of half a litre.

Separation with Soda Phosphate.

This reagent was proposed by Carnot (CHEM. NEWS, xlii., 85). He operates on a solution slightly acidified with acetic acid. After adding an excess of phosphate the mixture is boiled and filtered. Those results in the table obtained on "unneutralised" solutions are precisely after Carnot's instructions. The solutions contained 1 per cent acetic acid (33 per cent).

TABLE IX.

Saturated solution soda phosphate.	Percentage separation.	
	"Neutralised."	Not "neutralised."
20 c.c.	80.3	—
30 "	100.4	—
90 "	99.6	99.4
100 "	—	99.6
150 "	—	99.9

The 20 c.c. soda phosphate was unable to quite precipitate the half gram. of aluminum, although a clear solution was obtainable by repeated filtration.

These illustrations do not exhaust the number of methods which have been applied to the separation of the two bodies, but they sufficiently show that a compound so feeble as to be decomposed by merely washing may yet withstand the attack of strong precipitants.

In steel analysis only very small amounts of alumina and chromic acid come to be separated, and the defects are correspondingly insignificant. Even here, however, the use of ammonia and its carbonate in minimum quantities should be justified or abandoned: there is always a danger attending the general use of those methods whose range of accuracy is very small. Those methods, too, in which the aluminum salt is decomposed and precipitated by a neutral alkaline salt are open to suspicion. The method by Wöhler in which the mixed Al_2O_3 and Cr_2O_3 are dissolved in HCl, treated with excess of soda hydrate, oxidised with chlorine, and finally precipitating the still dissolved alumina with ammonium carbonate, belongs to the class already typified in Table VII. In face of the strong tendency to form a basic aluminium chromate, no process in which the aluminium is precipitated from neutral solution can be judged accurate until it has experimentally been proved to be so. Those processes in which the chromic acid is precipitated from the mixed solutions have not been studied.

The Separation of $Al_2O_3 + Fe_2O_3$ from CrO_3 .

It may be noticed that none of the methods observed both with iron and aluminum are equally satisfactory with either. The frequency with which these oxides are associated makes such a method desirable. Neither soda hydrate nor soda carbonate could be used except in conjunction with further means of separating the dissolved alumina. Soda phosphate, which serves so well for aluminum, does not give good results if the ferric solutions are "neutralised," and the presence of even considerable quantities of acetic acid is not a great improvement. An explanation of the different behaviour of neutralised solutions of iron and aluminum seems to lie in the fact that, while heating favours the formation of basic ferric chromates, it prevents the formation of the like aluminic compounds.

By ordering the solution so that only the free acid be neutralised and no dissolved ferric hydrate formed—this point is sufficiently indicated by the change of colour from yellow to reddish—the separations are accurate. This process tested with 0, 2, and 5 per cent of acetic acid present, and on the usual mixture of Fe_2Cl_6 and K_2CrO_4 , gave recoveries of 99.8, 99.9, and 99.6 per cent respectively.

Totley, near Sheffield.

ON NASCENT HYDROGEN.

By D. TOMMASI.

SOME years ago (*Comptes Rendus de l'Institut des Sciences de Milan*) I was occupied with the question as to whether the reducing properties peculiar to hydrogen, when set free from combination, are due to an allotropic state of the hydrogen, such as the nascent state, or to ordinary hydrogen under novel thermic conditions.

I studied the greater part of the reductions caused by hydrogen, which are generally but erroneously attributed to its nascent state, such as:—The reduction of the chloride, bromide, and iodide of silver; of chloric acid and the chlorates; of perchlorate of potassium; of the nitrates, &c.; and I here give in brief the results I obtained:—

1. Chloride of silver, in suspension in water acidulated with sulphuric acid, was treated with sodium amalgam. The experiment, which lasted forty minutes, was carried out in the dark. The chloride of silver remained perfectly white, and consequently had undergone no reduction; further, the liquid separated from the chloride of silver contained no trace of chloride of sodium. It is exactly the same with the bromide and the iodide of silver. On the other hand, a current of electricity will decompose the chloride of silver in suspension in acidulated water. In face of this, how can we explain why the chloride of silver, which resists the action of the nascent hydrogen produced by the decomposition of water by sodium amalgam, is reduced by the equally nascent hydrogen produced by the decomposition of the water by the electric current.

2. A saturated solution of chlorate of potassium was acidulated with sulphuric acid, then divided into two parts. One part was treated with zinc, the other with sodium amalgam; both reactions were stopped at the same time, while there was still some zinc and sodium amalgam left, and before the sulphuric acid was completely neutralised. The two solutions were filtered, and nitrate of silver was added. The solution which had been treated with the zinc gave a very abundant precipitate of chloride of silver, while the other solution, which had been treated with sodium amalgam, remained perfectly limpid. Chloric acid, and the chlorates of sodium, barium, copper, lead, and mercury undergo no reduction by sodium amalgam, whether we conduct the experiment in alkaline, neutral, or acid solution. Hydrogen formed by the electrolysis of water has no greater tendency to deoxidise chlorate of potassium.

3. A solution of perchlorate of potassium, chemically pure, was submitted to the action of various reducing agents, and the following results were obtained:—

(a). Perchlorate treated at the ordinary temperature, by zinc and dilute sulphuric acid, is not transformed into chloride, even when the solution is warmed.

(b). Magnesium and dilute sulphuric acid do not reduce the perchlorate, either hot or cold.

(c). A certain quantity of perchlorate was dissolved in a concentrated boiling solution of sulphate of copper; a piece of zinc was plunged into this,—immediately a violent reaction was produced, with a lively disengagement of hydrogen, but the perchlorate underwent no reduction. Neither is the perchlorate reduced when treated with

sodium amalgam, or with zinc and potash, warm, &c., or, in a word, by any of the principal reducing agents in ordinary use.

Now this perchlorate which has resisted all reduction, although in the presence of nascent hydrogen, is easily transformed into chloride by the action of a compound which does not give off hydrogen, viz., hydrosulphite of sodium. Although by the action of zinc on bisulphite of sodium (a reaction which gives birth to hydrosulphite of sodium) no hydrogen is given off; let us admit, for the sake of argument, that, on the contrary, there is. But then how could we explain that this same perchlorate, which experiences no reduction by nascent hydrogen in a large number of reactions, can be reduced by the hydrogen set free by the action of zinc on bisulphite of sodium? And why should nascent hydrogen, produced by the action of zinc on dilute sulphuric acid, reduce chlorate of potash and have no action on the perchlorate?

In these last two cases is it not always hydrogen in the nascent state which is given off? and the medium in which it is produced, is it not the same?

4. A solution of sulphate of nickel, even very dilute, to which potash and cyanide of potassium have been added, acquires a beautiful red tint by the action of zinc; at the same time hydrogen is given off. If in this experiment we replace the zinc by magnesium, or, better still, by the magnesium-platinum couple, we still notice the disengagement of hydrogen, but the red colouration is no longer produced. On the other hand, if the double cyanide of potassium and nickel is electrolysed, the red colour appears at the negative pole.

This must be due to one of two things:—either the red compound is produced by the metal or else by the nascent hydrogen.

In the former case the red tint should not be produced by the action of the hydrogen formed by the electrolysis of the water, and in the latter case it ought to be seen, since the hydrogen given off by the magnesium-platinum couple is just as nascent as that given off by the zinc or by the electric current.

5. S. Kern observed that, by causing magnesium to react on ferric chloride, ferric hydrate was produced. The note in which this observation was made was published by the *Bulletin de la Société Chimique de Paris* (vol. xxvii., p. 338), but the editor took care to add "but it does not appear probable that ferric hydrate should be produced, as there is a disengagement of hydrogen, or at least if there is no intervention of air."

I have repeated this experiment, taking care to keep all air away, and using only boiled water; but the results I obtained have always been identical with those of Kern. It is exactly the same if we substitute sodium amalgam for the magnesium.

6. According to the experiments of Stalschmidt, the nascent hydrogen produced by the decomposition of the water by powdered zinc transforms the nitrate of potassium into nitrite, the red cyanide into the yellow; it reduces the iodide and the iodates, but it does not reduce the chlorides.

7. De Wilde has stated that while sodium amalgam reduces bromate of potassium, it has no action on the chlorate.

In this latter case, as in the preceding one, is it not always nascent hydrogen which is given off?

If, then, the properties of nascent hydrogen were inherent to this allotropic state of the gas, we always ought to obtain the same reactions; but the experiments I have just quoted, and many others which I have passed over in silence, prove, on the contrary, that the reducing power of nascent hydrogen varies according to the chemical reaction which has produced it. And if this gas in the nascent state possesses a greater affinity than in the ordinary state, that is caused simply by the fact that the hydrogen, at the moment of being set free, is accompanied with all the heat which is produced while it is being set free.

Consequently, nascent hydrogen is synonymous with $H + \text{cal.}$, and the differences that we observe between hydrogen produced by different chemical reactions have their reason in that these reactions do not all give off the same quantity of heat.

In fact, if we represent nascent hydrogen by $H + \alpha$ (α being the quantity of heat given off by the chemical reaction which produces the hydrogen), we shall get for α the following values:—

Chemical reaction.	Heat produced (α).
$SO_4H_2 + Aq + Zn$	38°0 cala.
$SO_4H_2 + Aq + Cd$	23°8 "
$SO_4H_2 + Aq + Mg$	112°0 "
$2HCl + Aq + Zn$	34°2 "
$2HBr + Aq + Zn$	34°2 "
Sodium amalgam + Aq	112°0 "

As can be seen, the value of α varies with each chemical reaction, and consequently the hydrogen should be the more active as the value of α increases, bearing in mind that the reaction between the hydrogen and the substance to be reduced or to be hydrogenised may have commenced. (In a future note I shall show that without this clause the principle of maximum work is quite wrong). There are, however, some cases when the reduction is not due to $H + \text{cal.}$, but to the metal serving for the disengagement of hydrogen ($M + \text{cal.}$). Such is the case in the reduction of potassium chlorate by zinc and dilute sulphuric acid, or by the electrolysis of its solution using zinc as an anode.

In fact, when we electrolyse a solution of chlorate of potassium to which a few drops of sulphuric acid have been added, we obtain, according to the nature of the anode, an oxidation or a reduction of the salt.

Platinum Electrodes.—Perchloride is formed at the anode, without a trace of chloride at the cathode.

Platinum Cathode and Zinc Anode.—Chloride of potassium is formed only at the anode, with not a trace at the cathode.

The reduction of the chlorate in this case cannot be attributed to hydrogen, but to the zinc, which unites with the oxygen of the chlorate, forming oxide of zinc according to the equation $KClO_3 + 3Zn = KCl + 3ZnO$.—*Moniteur Scientifique*, Series 4, vol. xii., March, 1898.

ON A SIMPLE METHOD OF PREPARING HYDRONITRIC ACID.

By M. DENNSTEDT and W. GÜHLICH.

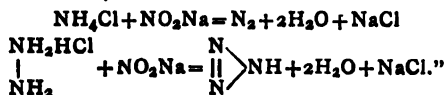
SOME time ago one of us put forward the opinion that the properties of argon, if we considered this gas as triatomic nitrogen, could be explained up to a certain point by admitting a central bond between the atoms of nitrogen. Although subsequent researches have left no doubt as to the elementary nature of argon, we nevertheless thought it interesting to submit our supposition, as far as could be done, to experimental verification. In hydronitric acid we have already a cyclic liaison of the atoms of nitrogen, and it seemed possible by carefully arranged oxidation to eliminate the atom of hydrogen, leaving intact the nucleus of three atoms of nitrogen.

Experiment has shown that the gas obtained by oxidising hydronitric acid by permanganate of potash in the presence of sulphuric acid in an atmosphere of carbonic anhydride is nitrogen, and not argon. The hydronitric acid necessary for this experiment was prepared by a very simple process, which enables one to obtain a dilute solution of the acid in any desired quantity in a few minutes. Admitting that the dilute acid can be easily concentrated by distillation, our process applies equally to the preparation of the concentrated acid. This process is very convenient as a lecture or class experi-

ment, as by its use we can prepare hydronitric acid from inorganic substances; it being a serious inconvenience to have to use, in the early lessons on inorganic chemistry, complex organic substances in order to prepare such a simple inorganic compound as hydronitric acid.

Curtius (*Berichte*, 1893, p. 1263; *Ibid.*, 1890, p. 3023) has already pointed out the possibility of obtaining nitramide by the action of nitric acid on hydrate of hydrazin, but this method has always given us very poor returns, so that we do not consider it advisable even for the preparation of small quantities of hydronitric acid.

At the commencement of his first memoir, Curtius says:—"In the same manner that the action of nitrous acid on ammonia gives rise to the formation of nitrogen and water, so should the action of the nitrites on monochlorhydrate of hydrazin give rise to the formation of hydronitric acid,—

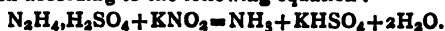


However, it appeared to be difficult to prepare hydronitric acid, starting directly from hydrazin and nitrous acid; in fact, under these conditions we were unable to obtain it.

The formula of the monosulphate of hydrazin is $(\text{NH}_2\text{NH}_2)_2\text{H}_2\text{SO}_4$. If we distil a mixture of monosulphate of hydrazin and nitrite of potash in aqueous solution, there passes—without any preliminary disengagement of gas—first a trace of hydrazin and no ammonia, after which the quantity of hydrazin increases, and at the same time a little ammonia comes over, and finally the hydrazin disappears entirely, to give place to the ammonia.

The monosulphate of hydrazin is thus decomposed under these conditions into hydrazin and ammonia without the formation of any hydronitric acid.

The bisulphate, however, behaves quite differently. Commercial sulphate of hydrazin is the secondary salt $(\text{NH}_2\text{NH}_2)_2\text{H}_2\text{SO}_4$. This salt should react with nitrite of potash according to the following equation:—



If to a solution of sulphate of hydrazin (5 grms.) we add a solution of nitrite of potassium (3.3 grms.) in about 200 c.c. of water, a lively disengagement of gas takes place, which only diminishes very slightly even when the solution is reduced to a very low temperature. We at first thought that this gas was nitrogen, resulting from the decomposition of the hydronitric acid formed; but later we satisfied ourselves that the reaction was effected in quite a different manner. If the liquid is distilled after the gas has ceased to come off a considerable quantity of hydronitric acid passes over. It is easy to concentrate the dilute acid by repeated distillations. The return is more satisfactory when the two solutions have been thoroughly cooled before mixing. Commercial nitrite of potassium contains a considerable quantity of free alkali, and it is of importance to exactly neutralise the alkalinity, or better still to add at once the quantity of sulphuric acid necessary to combine with the free alkali and set free the nitrous acid. The return obtained from 5 grms. of sulphate of hydrazin rises then to 0.2 grm. of N_2H_4 , while the disengagement of gas does not diminish to any appreciable extent. It results from this fact, that the disengagement of gas is not a secondary phenomenon, but constitutes an essential factor of the reaction. The analysis of the gas given off shows that it consists of nitrogen, oxygen, and protoxide of nitrogen. Although the quantitative analysis of a mixture having such a composition offers certain difficulties, we have succeeded in estimating the oxygen approximately, by absorbing it by pyrogallate of potash, the protoxide of nitrogen by repeated absorptions by water, and the nitrogen by difference.

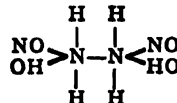
The nitrogen, we might mention, did not contain any argon. The results of our analyses have led us to believe that the formation of hydronitric acid takes place according to the following equation:—



According to this equation, 3 grms. of sulphate of hydrazin should furnish 1.1 grm. of hydronitric acid; but we were only able to obtain 0.2 grm., or 20 per cent of the theoretical quantity. Further, the volumes of the oxygen, the nitrogen, and the protoxide of nitrogen ought to be in the ratio 1:1:2. Now, we have always found twice the calculated volume of nitrogen, and a little less oxygen than there should be. This can only be accounted for on the supposition that a part of the oxygen set at liberty oxidises a part of the hydronitric acid formed into water and nitrogen. As for each volume of oxygen which takes part in the reaction, six volumes of nitrogen are formed, the result we have obtained is explained in a very simple manner. It is evident that the formation of hydronitric acid, starting from secondary sulphate of hydrazin, should follow quite a different path than that indicated by Curtius in his experiment with monochloride of hydrazin and nitrite of soda. In this reaction with three soda atoms, the hydronitric acid ought to be formed from the nitrogen of the hydrazin and that of the nitrous acid, while in our reaction the whole of the nitrogen of the nitrous acid is eliminated in the state of free nitrogen and protoxide of nitrogen; the nitrogen in the hydronitric acid formed being furnished exclusively by the hydrazin. We can illustrate this method of the formation of hydronitric acid in the following manner:—Sulphate of hydrazin and nitrous acid first gave rise to the formation of nitrite of hydrazin:—

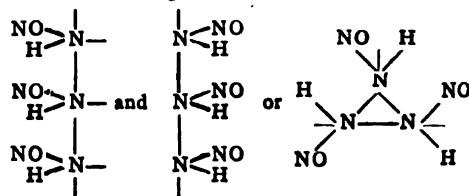


or—

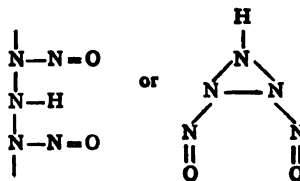


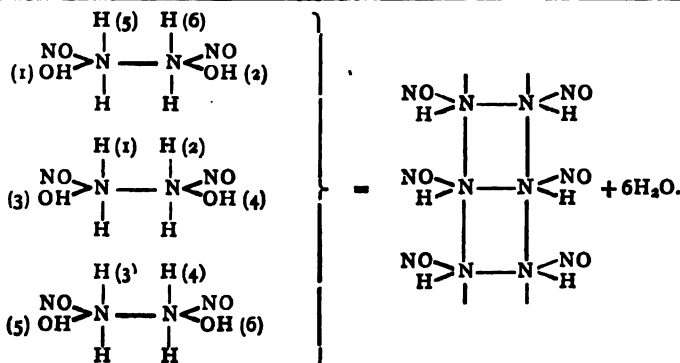
As with all the ammonium salts, the nitrogen is pentavalent in this compound. In the next phase, three molecules of the slightly stable nitrite of hydrazin form a cyclic compound by eliminating six molecules of water. The hydroxyl groups and the atoms of hydrogen which unite to form water are indicated in the formula by the same figures. (See formula, next page).

By breaking the bonds between the atoms of the hydrazinic nitrogen, two new molecules are formed—unsaturated, and therefore unstable—which already enclose three atoms of nitrogen re-united in a cyclic nucleus.

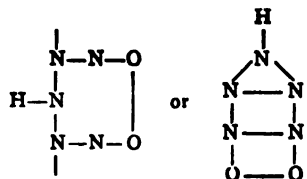


By the loss of one molecule of water (the oxygen is supplied by the group NO, nitrogen being given off at the same moment) there is formed—





This intermediate compound can also be expressed by the formula—



which takes account of the subsequent elimination of the protoxide of nitrogen and the oxygen. This elimination leads to the formation of hydronitric acid. It is well understood that the above phases may take place simultaneously.—*Chemiker Zeitung*, p. 876, 1897).

ON THE AMMONIA TEST OF COCAINUM HYDROCHLORICUM ACCORDING TO MACLAGAN.

By Messrs. C. F. BOEHRINGER and SOEHNE,
Waldhof, near Mannheim.

IN No. 1 of the *Pharmaceutische Centralhalle*, January 6, 1898, Dr. Fritz Günther inquired into MacLagan's ammonia test, the result of his investigation being:—

1. The occurrence of MacLagan's reaction depends on chance, and the test, therefore, is unreliable.
2. It is not known what impurities are to be demonstrated by this reaction.
3. It is established that there is no difference in physiological action between a cocaine fulfilling the requirements of the German Pharmacopœia III., which shows the reaction, and a cocaine which does not show it.

We consider it our duty to reply to this condemnation of MacLagan's test, the great value of which in testing commercial cocaine we have recognised for many years past, owing to the fact that a preparation not standing this test contains a larger amount of the admittedly toxic isotropylcocaine than should be permissible nowadays for medical use.

This circumstance, having long since come to the knowledge of the parties interested, it is all the more surprising to us how a cocainum hydrochloricum, containing a considerable percentage of isotropylcocaine, placed on the market by an otherwise renowned firm, could find a sale. We can explain this only by assuming that buyers relied on the manufacturer's good reputation, and that they were content with the fact of the preparation complying with the requirements of the Pharmacopœia. Thus the efforts of manufacturers to put within reach of medical men the purest possible preparations are practically useless, until

the Pharmacopœia Committee recognises that something better than hitherto admissible is within reach, and issue stricter regulations for testing the remedy concerned.

We will now try to prove the strength of the reasons in favour of MacLagan's test, and the weakness of the arguments adduced against this otherwise generally recognised test by Dr. Günther in favour of the inferior preparation.

Whereas MacLagan states that an amount of 4 per cent of by-alkaloid (when publishing his test in 1887 MacLagan did not know of the existence of isotropylcocaine) causes a milky turbidness of the liquid, manufacturers of cocaine are well aware of the fact that it is isotropylcocaine which in the ammonia test prevents a crystalline precipitate of the cocaine.

In order to demonstrate that this impurity exists in the above-mentioned commercial preparation, we indirectly bought $\frac{1}{2}$ kilogram. thereof and tested it.

One hundred grms. of this cocainum hydrochloricum were dissolved in water, precipitated by a solution of soda, and dried. The alkaloid was dissolved in absolute alcohol, the alcoholic lye, after cooling, was drawn off from the precipitated cocainum purum, and evaporated to dryness at a moderate heat.

By repeatedly treating it with petroleum ether (see Liebermann, "Report of the Deutsche Chem. Gesellschaft," 1888, p. 2343) we succeeded in isolating from the residue 3.6 grms. of an alkaloid very difficult of solution in petroleum ether. To demonstrate its really being isotropylcocaine, it was split up according to Liebermann's method, by concentrated hydrochloric acid ("Report of the Deutsche Chem. Gesellschaft," 1888, p. 2346), whereby we obtained 1.1 gm. of crude isotropa-acid, which, by treatment with a solution of barytes on the one hand, was converted into the readily soluble barytes salt of the γ -isotropa-acid; on the other hand, into the δ -isotropa-acid (the latter soluble with difficulty). After being but once re-crystallised from alcohol, the isolated acids from both salts showed the melting-points given by Liebermann, viz., 274° C. for the γ -acid and 206° C. for the δ -acid.

Thus it is proved that the cocaine in question contains at least 3.6 per cent of isotropylcocaine, probably more.

This cocaine does not stand MacLagan's test, and shows at once, on addition of the ammonia, a milky opalescent turbidness, indicating, according to MacLagan, a minimum amount of 4 per cent of by-alkaloid.

In order to ascertain more accurately the influence of the isotropylcocaine on MacLagan's test, we made up the following mixtures of pure hydrochlorate of cocaine* with hydrochlorate of isotropylcocaine,† 0.1 gm. to 87 c.c. of water, as we were accustomed to do, and tested each one after the addition of four drops of ammonia (sp.

* This cocaine stands MacLagan's test. After the addition of four drops (=0.2 c.c.) of ammonia (sp. gr. 0.95), followed by stirring for one and a half minutes, an intense crystalline precipitate took place, which pervaded the whole liquid.

† Obtained from the crude alkaloid by frequently boiling it with petrol ether.

gr. 0.96) as to its behaviour, vigorously stirring the liquid with a glass rod in a stout glass vessel, and pressing it from time to time against the walls of the vessel.

The following reactions ensued:—

	Per cent isatropylcocaine.		Minutes.	
1.	0.0	Crystalline precipitate after	1½	} Liquid clear at first.
2.	0.1	" "	2½	
3.	0.2	" "	5	
4.	0.4	Amorphous "	6	
5.	0.6	" "	8	
6.	0.8	" "	5	
7.	1.0	Hardly visible "	10	
8.	2.5	None.		
9.	3.5	" "		
10.	4.0	Liquid slightly opalescent.		
11.	5.0	" milky turbid.		
12.	10.0	" very "		

Accordingly, by means of MacLagan's test, a cocaine containing no isatropylcocaine or less than 0.2 per cent thereof, can be clearly distinguished from a more impure cocaine.

It is true that a cocaine containing 0.2 per cent of isatropylcocaine can hardly be distinguished from cocaine containing 1 per cent; hence the so-called irregularities exhibited in this test, for the very reason of its requiring a very pure cocaine. At all events, in this test a pure preparation must fulfil the requirements of showing in any case a considerable precipitate within from one to three minutes, perhaps after five minutes—dependent on the vigour in stirring.

If containing more than 1 per cent of isatropylcocaine—up to 4 per cent—the liquid remains clear on the addition of ammonia, but even after a quarter of an hour's vigorous stirring and pressing it does not show a crystalline precipitate worth mentioning. Not until the amount is 4 per cent does a slight opalescence appear after the addition of ammonia. With an amount of 5 per cent and more of isatropylcocaine the liquid becomes distinctly milky.

Regarding the size of the drops of ammonia, said by Dr. Günther to greatly influence the test, we beg to say:—For 0.1 grm. cocainum hydrochloricum there is theoretically necessary 0.05 grm. of an ammonia solution of 0.96 sp. gr. (containing 10 per cent of ammonia); 3 drops usually correspond to three times this quantity = 0.15 c.c. (20 drops = 1 c.c.).

This quantity, therefore, is sufficient, and the drops must be proportionately large.

According to the experiences we daily acquire regarding our own product, cocainum hydrochloricum, if containing less than 0.2 per cent of isatropylcocaine, stands MacLagan's test upon the addition of from 3 to 4 drops of ammonia. A considerable crystalline precipitate is deposited, which at the moment of forming pervades the whole liquid. (In the ammonia glass vessel generally used by us we found 4 drops to be equal to 0.2 c.c.).

According to "Commercial Organic Analysis" (Allen, vol. iii., Part 2, p. 280), MacLagan, moreover, suggests to take for 1 grain (= 0.0648 grm.) 2 drops of strong ammonia. By this none other can be meant than ammonia of a sp. gr. of 0.901 = 28 per cent, mentioned in the United States' Pharmacopœia under the heading of "Stronger Ammonia Water." Hence follows that for 0.1 grm. 3 drops of a 28 per cent ammonia solution should be taken,—therefore about 8 to 9 drops of a 10 per cent ammonia solution of a sp. gr. of 0.96, according to the German Pharmacopœia; whereas in our experience 0.2 c.c. of the 10 per cent ammonia solution is sufficient, that is, much less than mentioned by MacLagan. Sufficient ammonia, of course, to liberate the base must be present.

Summing up our opinion on MacLagan's test, we find that it very well admits of an accurate examination of cocainum hydrochloricum in reference to the amount of

isatropylcocaine, if the following points are strictly observed:—

1. For 0.1 grm. of cocainum hydrochloricum at least 0.15 to 0.2 c.c. of ammonia of a sp. gr. of 0.96 must be used.
2. Very vigorous stirring in a stout glass vessel is necessary, as, according to our observation, even in perfectly pure cocaine, artificially prepared from ecnogen, a precipitate is but rarely obtained without stirring. The walls of the glass vessel should be pressed with the glass rod immediately after the addition of ammonia, say ten times to and fro, to be followed by vigorous whipping with the glass rod.

If thus treated the precipitate is sure to take place in cocaine containing less than 0.2 per cent of isatropylcocaine.

We would still mention that the proportion of 0.1 grm. of cocainum hydrochloricum to 100 grms. of water, as stated by Dr. Günther, does not agree with the proportion mentioned by MacLagan. It is true that, owing to the confusing significations of English and American ounces, fluid ounces, avoirdupois and medicinal weight, it is not easy to get at the respective equivalents in metrical weight. A grain is always = 0.0648 grm.; 2 ounces of water calculated as ounces weight may be = 2×31.104 grms., in which case the proportion would be 0.0648 : 62.208 = 0.1 : 96.0; but if calculated on the basis of the commercial weight (1 ounce = 28.35 grms.), the proportion is 0.1 : 87.3.

Taking the weight of an American fluid ounce = 29.570 c.c. (of water therefore = 29.570 grms.) the proportion is 0.1 : 91.3. Anyhow, MacLagan's intention cannot have exceeded the proportion of 0.1 : 96.

We have found, on the one hand, that even in pure cocaine the precipitate occurs with greater difficulty if the quantity of water is increased, and, on the other hand, that by less stirring of the liquid with the glass rod it takes more than five minutes for the precipitate to occur, even with pure cocaine in a solution of 0.1 : 100.

Now, in order to bring about a practical and clear definition of MacLagan's test, we propose to dissolve 0.1 grm. of cocainum hydrochloricum in 85 c.c. of water, and to add as many drops of ammonia (sp. gr. 0.96) as correspond to 0.2 c.c.

As to the third point of Dr. Günther's condemnation of MacLagan's test, viz., the physiological effect of cocaine rendered impure by isatropylcocaine, we beg to say:—

There is no foundation for Dr. Günther's assertion that in respect of physiological action there is no difference between a cocaine standing MacLagan's test and a cocaine which does not stand it; in his publication even opinions of medical authorities are not quoted in demonstration thereof. No doubt, however, can exist as to the toxic effect of isatropylcocaine, for after its discovery Prof. O. Liebreich examined the alkaloid, and Prof. Liebermann ("Report of the Deutsche Chem. Gesellschaft," 1888, p. 2344), in his paper thereon, says:—

"Professor O. Liebreich was good enough to examine the alkaloid in reference to its physiological effect. According to his communication the alkaloid in question is of interest also, owing to its great toxic effect, and owing to its probably being the cause of the toxic secondary phenomena repeatedly observed after the administration of cocaine not quite pure. Its effect resembles neither that of cocaine nor of atropine; it is rather a *strong heart-poison*. A decrease in sensibility was neither observable when applied locally nor generally."

In spite of the toxic effect of this by-alkaloid being known, a preparation containing 3 to 4 per cent thereof is put on the market, bought unhesitatingly, and supplied to medical men! And withal a pure preparation, and the means of testing it are available.

A conscientious manufacturer of cocaine, therefore, should endeavour to supply a pure product. This is not requiring too much seeing that the best brands of this article are so supplied.

As shown by us, the test necessary for ascertaining the absence of this dangerous impurity has been furnished by MacLagan, and, indeed, in a thoroughly satisfactory form, so that everybody desirous of placing on the market medicaments of the greatest possible purity, is enabled to do so as regards cocaine.

By the foregoing we think we have demonstrated the necessity of submitting commercial cocaine to MacLagan's test.—*Pharmaceutische Centralhalle*, 1898, No. 9.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MARCH 31ST, 1898.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, April 9th, 1898.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 189 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from March 1st to March 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 189 samples examined by us, all were found to be clear, bright, and well filtered.

There has been a still further serious deficiency in the rainfall in the Thames valley during the past month. The actual amount which fell at Oxford was only 0.66 inch; the average for 30 years is 1.50 inches, leaving a deficiency of 0.84 inch, and bringing the present deficiency for the year up to 2.75 inches. The total rainfall for the first quarter of this year is only 2.67 inches, or less than half the thirty years' average.

Our bacteriological examinations of 268 samples have given the results recorded in the following table; we have also examined 37 other samples, from special wells, stand-pipes, &c., making a total of 305 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 27 samples) ..	814
New River, filtered (mean of 27 samples) ..	28
Thames, unfiltered (mean of 27 samples) ..	3917
Thames water, from the clear water wells of five Thames-derived supplies (mean of 133 samples) ..	25
Ditto ditto highest	296
Ditto ditto lowest	0
River Lea, unfiltered (mean of 24 samples) ..	1193
River Lea, from the East London Company's clear water well (mean of 27 samples) ..	24

The exceptionally dry weather is having the usual effect of keeping the river in good condition; this, in conjunction with the high efficiency of the filtering arrangements of the various companies, accounts for the purity of the water at present supplied to London.

The following comparison illustrates the improvement

in chemical composition consequent on an abnormally low rainfall. The rainfall in March, 1897, was above the average:—

Comparison of the Averages of the Five Thames-derived Supplies for the Months of March, 1897 and 1898.

Common Salt. Per gall.	Nitric Acid. Per gall.	Hardness. Degrees. gall.	Oxygen. reqd. Per gall.	Organic Carbon. Per gall.	Carbon. Per gall.	Colour. Per B'n: Blue.
Means.	Means.	Means.	Means.	Means.	Max.	Means.
March, 1897. 2'018	1'035	17'12	0'064	0'152	0'196	17'2: 20
1898. 2'167	0'877	16'06	0'033	0'076	0'109	10'9: 20

We are, Sir,
Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

THE DIGNITY OF ANALYTICAL WORK.*

By C. B. DUDLEY.

(Continued from p. 174).

ANOTHER illustration from a different field will emphasise the power of an analysis to explain difficulties. A lot of boiler plate was at one time received at Altoona shops, from one of the best makers. In this lot of forty or fifty sheets, two were found which gave difficulty in flanging, this operation consisting, as is well known, in bending over the edges of the sheets while hot, nearly at right angles to the balance of the sheet, in order to enable it to be joined to other sheets in the boiler. The two sheets referred to cracked in the bend, although the remainder of the lot gave no difficulty from this cause. The workmen being thoroughly experienced, and the practices of the shop being excellent, the cause of the failure in the case of these two sheets was not apparent. An analysis of samples from each of these sheets, however, showed 0.35 per cent and 0.36 per cent of carbon respectively, while analyses of samples from other sheets in the same lot showed in no case above 0.12 to 0.15 per cent of carbon. The explanation of the difficulty seemed now quite clear. The shops had been supplied for a long time with the softer grade of steel, and the methods and practices in use were those applicable to that kind of steel. No wonder, then, that with the harder grade difficulty should arise, as actually happened, and but for the analysis this might have passed into shop traditions as one of those unexplained and unexplainable crochets of steel which both the makers and practical users of this metal delight in constantly bringing forward.

A single illustration further will, perhaps, suffice on this head. A few years ago a shipment of some three hundred freight axles was received at two different shops on the Pennsylvania Railroad, from an entirely reputable maker. Some of these axles were used for repairs, and some went under new cars. Scarcely had they gotten into service, however, before difficulty began to arise. The axles began to break; indeed one of them broke before the car had been turned out of the shop yard, one broke into three pieces before the car had made 150 miles. and in less than three months eight had broken. Each of the broken axles was sent to the laboratory, and a careful study of the case made, with the hope of discovering the cause of the failure. An examination of the freshly fractured ends of several of the broken axles showed that for a little distance in from the circumference the fractured steel presented an appearance quite different from that given by the remainder of the fracture. Moreover, a line of demarcation between these two apparently different kinds

* Presidential Address delivered at the Washington Meeting of the American Chemical Society, December 29, 1897. From the *Journal of the American Chemical Society*, vol. xx., No. 2.

of steel in the same axle could be clearly traced. Accordingly it was decided to make analyses of borings from near the circumference and near the centre, and see whether this would reveal anything. It may be stated that the axles were known to have been made from Bessemer steel, and should normally have contained not more than 0.10 per cent of phosphorus. The analysis of the borings from near the circumference of the axles in no case gave figures up to this limit, while the borings from the centre of the axles in no case showed less than 0.16 per cent phosphorus, and in some cases the amount was as high as 0.24 per cent. Those who are familiar with the methods in daily use in modern steel works will from these figures at once understand the cause of breakage of these axles. For the benefit of those who are not, it may be well to explain that in most modern steel works large ingots are now the rule, and that in large ingots which take considerable time to solidify from the molten condition analyses show that some of the constituents of the steel are not uniformly disseminated throughout the mass. This separation of the constituents during cooling, technically known as "segregation," is characteristic of the carbon, the phosphorus, and the sulphur. Furthermore, the segregation appears to be worst in the upper third of the ingot, so much so that many specifications now require the upper third of the ingot to be removed, and not used at all in making the articles the specifications call for. This much being stated, it is clear why our axles broke. They were made from badly segregated steel, perhaps from the rejected upper thirds of a lot of ingots the balance of which were used for other purposes. Subsequent correspondence with the parties furnishing the axles gave good grounds for belief that such was the case. For the comfort of those who ride on railroads it may be added that the three hundred axles were at once withdrawn from service, and that since that time a chemical and physical specification for both passenger and freight axles has been prepared, which is believed to preclude the possibility of such axles as are described above being received by the Pennsylvania Railroad.

These illustrations of the power of an analysis to explain difficulties could be prolonged to almost any extent, but I spare you. Furthermore, I should not like to be understood as claiming that every puzzle, every difficulty, or every state of affairs in Nature, where the reasons for the phenomena which we find are not apparent at sight, can be explained by a chemical analysis. Our knowledge is far too limited for this. Moreover, many cases could be cited in which an analysis throws no light whatever on the situation; but notwithstanding this, an experience of some twenty years in seeking out the causes of things, as a necessary preliminary to the intelligent modification of practices and methods, in connection with a great corporation, has continually impressed me more and more with the very great help which a properly conceived and executed analysis can give in cases of difficulty.

But again I take pride in the field of analytical work, because of the opportunity which thoughtful analytical work affords for finding new things. The careful, thoughtful, observant analyst is constantly on the verge of either being able to add to his own knowledge, or of being able to contribute something to the general progress of our science. And here again I must be pardoned for using, as illustrations, cases which have arisen in the laboratory of the Pennsylvania Railroad Company.

A few years ago, in our laboratory, we began to get ready to make our analyses of the samples of steel which were designed ultimately to be the international standards for the analysis of iron and steel. Before starting in on these samples, however, it was deemed prudent to do a little preliminary work on some other samples, with the idea in mind of seeing whether apparatus and method were satisfactory. Accordingly four separate and distinct determinations on the same sample were made for carbon,

using the double chloride of copper and ammonium to release the carbon, and burning in oxygen gas. The four determinations agreed with each other within 0.01 or 0.02 of a per cent, and were regarded as fairly satisfactory. But as the work was important, and as some parts of the apparatus had not worked quite satisfactorily, it was decided to repeat the four determinations. Meanwhile a new stock bottle of solution of the double chloride had been made exactly in the manner that had been our custom for some time previous. When the second four determinations were obtained, they differed from the first by more than a tenth of a per cent. I need not weary you with the details of our hunt for the cause of this discrepancy,—how every point in the apparatus was tested up one after another, how various modifications were tried, how combustions were made on crystallised sugar to check ourselves, and how finally we located the difficulty in the double chloride of copper and ammonium solution. These details have all been published (*Trans. Amer. Inst. of Mining Engineers*, xix., 614). Suffice it to say that as the result of this work, together with subsequent work by other chemists, it is, we believe, now generally accepted that the commercial ammonium double salt contains carbon in some form, probably pyridine, that its use as a solvent to release the carbon from iron and steel is unreliable, and that the substitution of the potassium for the ammonium double salt overcomes these difficulties. The point which I especially want to emphasise is, that in trying to do a little careful analytical work we struck a new and apparently hitherto unsuspected source of error in one of the oldest and best established methods of iron and steel analysis.

Another illustration will perhaps make this point still more clear. In the regular course of work at one time a silicon determination was made in a piece of tire steel, which had been sent by an officer of another railroad, for information. The figures found by our analysis were 0.14 per cent, these figures being sent to the officer above referred to. A little later, word was received that an analysis of a sample from the same tire, made by another chemist, gave 0.28 per cent as the content of silicon. This, of course, led us to look over our work, with the idea of finding where the cause of the discrepancy lay. A careful examination of our weights and figures showed that it was not an error of calculation. Accordingly we decided to duplicate our work, need I say with the expectation of finding that the other chemist had made a mistake? Judge of our surprise when we found that our second analysis confirmed his figures exactly. Our first and second analyses had been made by the same method, and by the same operator, working on borings from the same bottle, and the cause of the discrepancy between the two was not, therefore, at first sight apparent. On carefully questioning the operator, however, as to exactly what he did at each step of the method, a clue was obtained, which, when followed out, cleared up the whole difficulty, and ultimately led to a modification of the method. The silicon in these samples was determined by what is known as Drown's method, consisting in dissolving the steel in nitric acid, adding sulphuric, heating until white fumes of the latter acid appear, to render the silica insoluble, dilution with water, filtration, washing, and weighing. The difference between our two analyses consisted simply in this—that in the first case, after the dilution with water, there being considerable work in hand, the vessel was allowed to stand over night before filtration, while in the second case filtration immediately followed dilution. Subsequent work on this point showed that in this method silica is not completely dehydrated by heating in concentrated sulphuric acid, in presence of iron salts, but is apparently rendered colloidal and sufficiently dehydrated, so that, if filtration follows soon after dilution, fairly accurate results will be obtained. On standing after dilution, however, this colloidal, un-dehydrated silica apparently goes into solution again. Indeed we were able to get on this same sample,

anywhere from one-eighth up to the full amount of silicon present, by varying the time of standing after dilution, the longest time covered by our experiments being about four days.

(To be continued).

CORRESPONDENCE.

VOLUMETRIC DETERMINATION OF CHROMIUM IN IRON AND STEEL.

To the Editor of the Chemical News.

SIR,—In Mr. H. Brearley's article on "Separations from Chromic Acid" in your issue of March 25th (CHEM. NEWS, vol. lxxvii., p. 131), he draws attention to some possible errors in the volumetric determination of chromium in iron and steel as described by me in 1877 in the CHEMICAL NEWS (vol. xxv., p. 151), and in 1893 before the Iron and Steel Institution, for the consideration of which I would crave a little space.

The criticism is somewhat curious, and is, I think, due to a good deal of misapprehension. I do not know what objection Mr. Brearley refers to when he says "Galbraith deals with some objections which had been repeatedly urged against his method."

Referring to my paper of 1893, Mr. Brearley says:—"He might have reduced the needlessly large bulk of oxides by adding less permanganate, a procedure we had long before found to minimise the error." If he (who does Mr. Brearley mean by "we"?) found this out he has carefully kept it to himself (I knew it in 1878), but I am not aware of any explanation of it being given before that given by Mr. Stead and myself in 1893.

Again, he says—"but nowhere does he adopt the more satisfactory course of dissolving a pure carbon steel along with a known quantity of a chromium salt." I say in the paper—"I now tried the effect of the presence of iron," but this is without the use of soda. The experiments, as recorded below, fulfil those conditions, but there is no reason to suppose, unless Mr. Brearley's objection is correct, that the testing of it with a solution of chromic acid or bichromate solution was not a fair method of testing it, supplementing these with examples on actual chromium iron.

Let me remind Mr. Brearley, however, that the title of my paper in 1893 was "The Determination of Chromium in *Ferro-chromium*." I nowhere recommend the use of soda for chromium steel; it is not necessary, and quite accurate results can be got without it.

I regret that Mr. Brearley's paper is full of criticisms of the above character, because it is apt to disguise the fact that he has hit upon quite a likely source of error, and his paper otherwise is valuable.

His objection is that when soda is added to the sulphuric acid solution of chromic oxide in presence of the manganese salt got from the reduction of the added permanganate, the precipitated oxide of manganese reduces the chromic oxide.

I think he gives away most of his case, however, when he says that "when he reduces a standard solution of bichromate and treats it with potassium permanganate and soda, he gets fair results." And, moreover, when he adds that he has "made trials on artificial and actual examples of chrome steel, and have obtained results in keeping with above explanation," I might reply that he ought to have given the figures, and further, that the modification, as I have already explained, was not meant for chromium steel.

I have carried out the following experiments to ascertain if there is any reducing action such as he describes.

Fifty c.c. standard bichromate solution was made up to 200 c.c., and reduced with SO_2 . Ten c.c. H_2SO_4 and

1 gm. $\text{K}_2\text{Mn}_2\text{O}_8$ was then added, and the solution boiled till the $\text{K}_2\text{Mn}_2\text{O}_8$ was entirely reduced. The solution was then diluted to about 250 c.c. and made alkaline with NaHO . It was then filtered and the insoluble matter washed, the filtrate made acid with HCl , 4 grms. $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$ added, and the excess of Fe as FeO determined with bichromate.

As 1 gm. of the iron salt took .. 14.3 c.c. $\text{K}_2\text{Cr}_2\text{O}_7$

4 grms. are equal to 57.2 " "

Less 50 c.c. used 50.0 " "

7.2 " "

The excess of iron took exactly 7.2 c.c. $\text{K}_2\text{Cr}_2\text{O}_7$. So far Mr. Brearley and I agree.

The above was repeated, excepting that 1 gm. soft steel was dissolved in sulphuric acid solution containing the bichromate, thus avoiding the reduction with SO_2 and fulfilling the conditions referred to by Mr. Brearley.

This also took exactly 7.2 c.c., and it corresponds with a ferro-chromium of 13.42 per cent chromium. As I felt that the reducing action referred to by Mr. Brearley might take place if it were given more time, I repeated the above experiment and allowed the soda solution to stand all night. I might say there is no excuse for this in actual practice, because the solution filters remarkably quickly. The bichromate solution taken was 8.4 c.c., equal to a loss of 1.2 c.c., or 0.37 per cent chromium.

The above were repeated and verified by our Mr. Macgregor, who got exactly the same figures.

I think the above fairly dispose of the objections raised by Mr. Brearley, and I think he will find on repeating them, or carrying out similar experiments, that the objection does not apply in the case of a chromium determination.—I am, &c.,

W. GALERAITH.

C/o Glasgow Iron and Steel Co., Ltd.,
Wishaw.

NOTICES OF BOOKS.

An Elementary Treatise on the Metric System of Weights and Measures. By J. HAMBLIN SMITH, M.A. London, New York, and Bombay: Longmans, Green, and Co. 1897. Pp. 103.

In 1895 a Parliamentary Committee recommended that the metrical system of weights and measures should be at once legalised for all purposes: this was accepted by Parliament, and an Act has been passed to legalise the metric system for the purposes of trade. The Committee also made other recommendations, which, however, were not agreed to. There can be little doubt that the metric system is bound to come in time; in all matters of scientific, *i.e.*, chemical, physical, engineering, &c., calculations, no one would think of using fractions, especially such barbarous fractions as, say, 1 lb. 10 ozs. of mutton at 10½d. a lb.

In the handy volume now before us the author explains and teaches in a most clear and succinct manner the simplicities of the decimal system. A number of problems are set throughout the book, and the correct answers are to be found at the end.

Sugar, Home-grown and Home-manufactured. By SIGMUND STEIN. Liverpool: C. Tinsling and Co. 1898. Pp. 71. Illustrated with 19 Engravings.

THE author describes this pamphlet as "a proof that one-and-a-half million tons of sugar consumed annually can be produced at home, and that about £15,000,000 per annum now paid for sugar from abroad can be used to support home industries and save the British farmer from ruin."

The manufacture of beetroot sugar dates from the last century. Marggraf first found that the root contained sugar in 1747, but it was first turned to practical account by Achard some years later, and in the year 1838 there were no fewer than 159 factories at work in different parts of Germany. The French next took the matter up, with the result that at the present day there are nearly 300 beetroot sugar-factories at work in France.

In the year 1853 a factory was started at Mount Mellick, in Queen's County, but it was an utter failure. Another factory was started at Lavenham, in Suffolk, but this failed also. The cause of these disasters is not far to seek, and with proper care and knowledge there is no reason why it should be repeated.

The climate of England is stated by many well-known chemists who have studied the question during the last fifty years to be quite as favourable—if not more so—to the growth of the sugar beet than that of the Continent. Full details are here given as to the necessary character of, and the best method of preparing, the soil intended for growing sugar beet; also detailed instructions as to manuring, sowing, and special cultivation: good cultivators get 14 to 16 tons of roots per acre, giving 12·7 to 16·9 per cent of sugar.

It must be remembered that beetroot takes very large quantities of different plant-foods from the soil, which must of necessity be returned to it; for instance, 100 lbs. of roots and 100 lbs. of leaves take away 43·8 lbs. of nitrogen, magnesia, phosphoric acid, potassium, and lime in varying proportions. The author then gives a lot of figures showing that, under fair conditions, beets can be grown in the United Kingdom at a good profit. Of course a factory must be erected to manufacture the sugar; but Mr. Stein shows (and he has had a great deal of experience) that, on a capital outlay of £60,000, a profit of over 6 per cent can be easily made, taking sugar at the low price of £9 per ton; £12 per ton would give 32 per cent profit.

A good description of the process of manufacturing raw sugar as practised at the present day then follows, with a number of excellent engravings showing the machinery used; the pamphlet closes with a number of tables and statistics concerning foreign factories.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 11, March 14, 1898.

Chemical Actions of the Electric Discharge. Nitro-compounds in the presence of Free Nitrogen.—M. Berthelot.—The principal nitro-compounds experimented on embrace examples taken from the fundamental classes, such as the mono- and poly-amines and the primary, secondary, and tertiary alkalis belonging to the fatty, benzenic, and pyridic series, &c. A large number of results are given, but the paper is far too lengthy for useful abstraction.

Estimation of Carbonic Oxide in Large Quantities of Air.—Armand Gautier.—The author refers to a paper in the last number of the *Comptes Rendus* by M. Nicloux, and claims to have used almost the same method for the past seven or eight years.

Causes of the Reciprocal Displacement of two Acids.—A. Colson.—When a metallic chloride is attacked by a fixed acid, hydrochloric acid gas is given off; while, on the contrary, sulphydric gas is absorbed by pure and dry metallic salts. The first reaction is endothermic, and the second strongly exothermic, but in both cases an increase of temperature assists the reaction, while a lowering of the temperature will retard or even stop it entirely.

New Silicide of Chromium.—Ch. Zettel.—The author has prepared a new silicide of chromium by adding 200 grms. of calcined sesquioxide of chromium to a melted bath of 140 grms. each of copper and aluminium in a fire-clay crucible. On pouring, a metallic ingot is obtained, containing a well-crystallised body, composed of silicon and chromium, having the formula SiCr_3 . It can be separated from the metals by means of aqua regia. After drying, it is in the form of a grey crystalline powder. Its density at $+18^\circ$ is 6·52. It easily scratches glass, but will not touch quartz; while the silicide obtained by M. Moissan, viz., SiCr_2 , will scratch even corundum with ease. It is soluble in hydrofluoric acid, but neither hydrochloric or nitric acid or aqua regia have any effect on it. The following figures have been obtained by analysis:—

	I.	II.	III.	Theory for SiCr_2
Si . . .	15·40	—	15·52	15·21
Cr . . .	83·81	84·68	84·98	84·79

New Method of Fractionation of the Yttria Earths.—G. Urbain.—Already inserted in full.

Two Methods for the Decomposition of some Sulphocyanic Ethers.—G. Chaner de Coninck.—The author has studied the action of hypochlorites containing an excess of alkali on four sulphocyanic ethers, viz., the sulphocyanates of methyl, ethyl, amyl, and methylene. His results are here described in full.

On some Ether Oxides of β -Naphthol.—F. Bodroux.—Several ether oxides of β -naphthol have been prepared by the action of a fatty bromide or iodide on naphthol- β dissolved in alcoholic potash. The mixture was heated for an hour on a water-bath. As soon as it reached the boiling-point the reaction took place; the colour of the solution changed from green to yellow, while at the same time a white crystalline deposit of bromide or iodide of potassium was formed round the sides of the flask. After cooling, the liquid was decanted and the alcohol driven off by heat; what remained was treated with an excess of potash. The phenolic ether is precipitated and floats on the surface of the liquid; it sometimes solidifies on contact with the air, and can be easily separated.

Sterilisation of Liquids by Filtration.—J. Haussner.—The author has found that of all the substances he has tried for the purification of water by filtration, the infusorial earth called "kieselguhr" has given the best results. It is prepared by first finely sifting, to remove all large particles, and then heating to about 800° or 1000° . On cooling, it must be powdered and levigated to obtain an impalpable powder—though this last operation is only necessary when certain special solutions have to be dealt with, experience alone showing when. To use it, the powder is mixed with a little water and thrown on a filter; as the liquid passes through, the powder remains, and forms a perfect layer for filtration and sterilisation.

Journal für Praktische Chemie,
1898, No. 2.

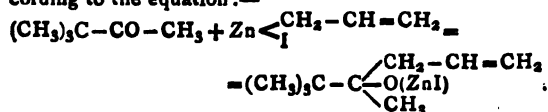
β -Naphthoquinolines.—Ad. Claus and H. Bessler.—This substance was prepared by Skrap's method, which gave a good yield when enough concentrated glycerin was used and the temperature favourable (135 — 145° is the best). After heating the whole on an oil-bath for about four hours, it was poured into ice-cold water and saturated with alkali. The raw product obtained was fractionally distilled; the fraction coming over at 340° consisting chiefly of naphthoquinoline. The base was purified by dissolving it in the least possible amount of sulphuric acid hydrate, and pouring the cold solution into alcohol. The sulphate separated in pale green crystals, and was decomposed with soda, and the base obtained (m. p. 89 — 90°). The analysis of this salt corresponded to the formula $\text{C}_{13}\text{H}_9\text{N}$. From this base various salts were prepared: β naphthoquinoline, iodmethylete, chlor-methylete, bichromate, methylete, and sulphomethylete.

α - or (1)-Naphthoquinolines.—Ad. Claus and P. Imhoff.—This substance is much more difficult to prepare than the β -naphthoquinoline. The authors find that the best method of preparation is to mix equal quantities of naphthylamine and arsenious acid in the finest powder, then add the glycerin, and finally the sulphuric acid hydrate, whilst the vessel is well shaken. The whole is then heated for some time at 100° and still kept well shaken until it becomes a light brown solution. It can then be heated to 150°, when the final decomposition takes place. The end of the reaction is known when a portion of the substance on dilution gives an orange-coloured precipitate with potassium permanganate, and the liquid is not coloured blue. α -Naphthoquinoline is insoluble in water, but soluble in ether, alcohol, benzene, acetic acid, and carbon disulphide. It melts at 51° and boils at 338°; not far above its melting-point it decomposes. The authors also prepared various salts from the free base.

Structural Relation between the two Naphthoquinolines.—Ad. Claus.—This paper is a discussion on the structural formulae for the naphthoquinolines, and is unsuitable for abstraction.

Para-pseudopropynaphthenic Acid.—W. Markownikoff.—This acid is prepared from cuminic acid by hydrogenation. Experience proves that twice acting with sodium in boiling amyl alcohol solution is enough, and a product is obtained free from cuminic acid, but many products of incomplete hydrogenation are formed, and these must be got rid of by the action of manganate solution. A substance is obtained of constant melting-point (94–95°) after re-crystallisation from hot alcohol. The substance, on combustion, is found to correspond to the formula $C_{10}H_{18}O_2$. Various salts of the acid are also prepared.

Allylmethyltertiarybutylcarbinol.—Al. Gnedin.—The author investigates the reaction of iodallyl and zinc on the tertiary butylmethylketones, assuming that the reaction is analogous to the syntheses of other unsaturated alcohols of the general formula $C_nH_{2n-1}OH$, according to the equation:—



The ketone and iodallyl are dropped on zinc and cooled with ice, and left standing for some time. It can then be worked up with water and distilled; the purest fraction coming over at 165–168°. It is a light yellow mobile liquid with a smell of camphor, solidifying at –7°. This substance can be oxidised to the corresponding glycerin with permanganate; the glycerin being crystallised many times from ether (m. p. 87–88°).

Unsaturated Hydrocarbons.—R. Walther.—When benzyliocyanide and benzaldehyde are heated to a temperature above 200°, not only is water split off, but a secondary action takes place: saponifying of the cyanogen group and formation of CO_2 . In a similar manner, when benzaldehyde and phenylacetic acid are heated under pressure, stilbene is formed. The temperature of maximum stilbene formation appears to be 250°. On cooling the mixture, the stilbene partially separates out in large crystals. Salicylic aldehyde and cuminic aldehyde react in an analogous way.

Zeitschrift für Physikalische Chemie,
Vol. xiv., No. 1.

Experimental Investigation of the Dissociation of Substances Dissolved in Aqueous Alcohol.—Ernst Cohen.—The substances were examined with regard to their dissociation, both when dissolved in pure alcohol and in aqueous alcohol; the alcohol content varying from 0–100 per cent. The experiments took place at 18°. The salts used were KCl, KI, KCl, NaCl, HCl. As a re-

sult of these experiments it was found that—(1) in dilute aqueous alcohol solutions the influence of the alcohol content on the molecular conductivity is independent of the concentration of the solution; (2) the molecular conductivity of an aqueous alcohol solution can be deduced from the equivalent water solution through multiplication by a factor which is constant for each alcohol content.

Employment of Metallic Sulphides as Electrodes.—Isidor Bernfeld.—In the electrical separation of Cu from its ores, the Cu is first washed to free it from all impurities except the noble metals, and is then used as an anode. By this means pure Cu is deposited on the cathode, while the other metals fall to the bottom. Lately it has been suggested that raw galena might be used as anode, but the method has been given up owing to the presence of various impurities in the ore. The author has investigated in this connection the phenomena attending the employment of galena and other metallic sulphides, both as anode and cathode. He finds that as anode in an acid cell the metal goes into solution and sulphur is deposited; with greater current density the S is oxidised. As cathode in an acid cell, H_2S results, and the metal remains behind. As anode in alkaline solution, the metal is changed to hydroxide, and the S oxidised; while as cathode in alkaline solution, alkali sulphide is formed and the metal remains behind.

Dissociation in Mixed Salt Solutions.—A. Fock.—The author shows that, on the assumption that in a mixed solution of two binary electrolytes with a common ion, the degree of dissociation of both salts is not equal, nor equal to that of the entire concentration. His former results on the mixed crystals of ammonium chloride and potassium chloride are in entire conformity with the distribution law and the law of mass action.

Universal Applicability of the Law of Dilution.—J. J. van Laar.—As is well known, the law discovered by Ostwald for weak electrolytes does not hold for substances which, like HCl, are to a great extent dissociated in aqueous solution. The author attempts to explain this by an assumption, which is supported by several arguments, that the electrical conductivity of a good electrolyte is not an exact measure of its degree of dissociation, e.g., assuming that the dilution law does hold, the calculated heat of solution of Aq acetate is 4369 g. cal.; of Aq propionate, 3789 g. cal.; and of Aq isobutyrate, 2715 g. cal. Whereas the numbers found by Fr. von Maarseveen were 4613, 3980, and 2860 g. cal. respectively; the error in each case being only about 5 per cent, although the degree of dissociation employed in the calculation is that determined by conductivity. The author believes that the degree of dissociation in a good electrolyte is modified by the passage of the current, in that the temperature of its ions and molecules is much higher than that of the surrounding medium.

Relationship between Heat of Solution, Solubility, and Degree of Dissociation.—Heinrich Goldschmidt.—The substances used were silver acetate, silver isobutyrate, silver propionate, and *o*-nitrobenzoic acid. The dissociation was found for saturated solutions at 25°. The heat of solution was measured in a Pt calorimeter, a solution of $AgNO_3$ being first added, and enough of a sodium salt to form the required salt, and the rise of temperature measured. The results of the experiment were compared with the calculated results according to van't Hoff's and van Laar's formulae. In the case of the silver salts, neither formula gave good results.

Equilibrium between Ammonium Nitrate and Ammonia.—B. Kuriloff.—Ammonium nitrate is only in a position to give a single compound by the absorption of dry ammonia; the composition probably being $NH_4NO_3 \cdot 3NH_3$, and the range over which this compound can exist lies at a very low temperature—from –40° onwards. At higher temperatures, until we reach the melting-point of ammonium nitrate, there is only the phase, solid ammonium nitrate.

Separation of Helium from a Natural Compound with strong Development of Light and Heat.—Julius Thomsen.—This mineral consists chiefly of fluorspar, but has a small percentage of fluo-compounds of Ce and Yt group. When powdered and heated, light and heat are developed, and it glows with a gold-coloured light. A gas is given off which, by the spectroscope can be recognised as helium.

Passage of Electric Currents in Mixed Solutions of Electrolytes.—K. Hopfgartner.—Mixtures of normal solutions of NaCl and HCl, BaCl₂ and HCl, and MgSO₄ and CuSO₄ were employed for this research. The chlorine was estimated with silver. The experimental results agree very well with the calculated ones, except in two experiments with MgSO₄ and CuSO₄.

Dissociation of Dibasic Organic Acids. Part I.—W. A. Smith.

MEETINGS FOR THE WEEK.

MONDAY, 25th.—Society of Arts, 8. (Cantor Lectures). "Sources of Commercial Indiarubber," by Dr. D. Morris, C.M.G.

TUESDAY, 26th.—Royal Institution, 3. "Phases of Art—Past and Present," by T. C. Gotch.

WEDNESDAY, 27th.—Society of Arts, 8. "Photography and Colour Printing," by Captain W. de W. Abney, C.B.

THURSDAY, 28th.—Society of Arts, 4.30. "India and Her Currency," by Sir Edward Sassoon, Bart.
— Royal Institution, 3. "Some Leaders in the Poetic Revival of 1760-1820—Cowper, Burns, and Scott," by The Rev. Canon Ainger, M.A., LL.D.

FRIDAY, 29th.—Royal Institution, 9. "Magneto-optic Rotation and its Explanation by a Gyrostatic Medium" (with Experimental Illustrations), by Professor Andrew Gray, M.A., LL.D., F.R.S.

SATURDAY, 30th.—Royal Institution, 3. "Programme Music" (with Musical Illustrations), by Sir Walter Parratt, Mus. Doc., Master of the Queen's Music.

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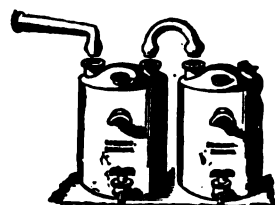
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PAGE

ARTICLES.—	PAGE
Note on a Curious Lead Salt.—The Double Iodide of Lead and Potassium, by Frederick C. Huxley Brooks	191
On the Decomposition of Concentrated Sulphuric Acid by Mercury at Ordinary Temperatures, by Charles Baskerville and F. W. Miller	192
Metallurgical Notes, by Sergius Kern, M.E.	192
Note on some Remarkable Thermo-effects produced under the Influence of High Pressure, by H. N. Warren	192
On Neodymium, by O. Boudouard	193
On the Yttric Earths contained in the Monazite Sands, by P. Schützenberger and O. Boudouard	193
The Dignity of Analytical Work, by C. B. Dudley	195
PROCEEDINGS OF SOCIETIES—	
CHEMICAL SOCIETY.....	197
PHYSICAL SOCIETY.....	199
NOTICES OF BOOKS.—The Tutorial Chemistry	200
CHEMICAL NOTICES FROM FOREIGN SOURCES	200
MEETINGS FOR THE WEEK	201

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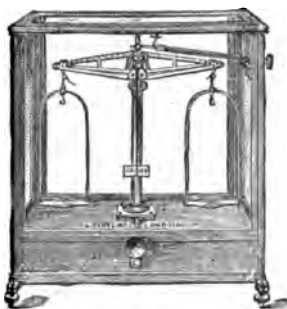
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THE CHEMICAL NEWS.

Vol. LXXVII., No. 2005.

NOTE ON A CURIOUS LEAD SALT. THE DOUBLE IODIDE OF LEAD AND POTASSIUM, $PbI_2 \cdot 2KI$.

By FREDERICK C. HUXLEY BROOKS.

EARLY in 1897, while testing a sample of zinc shavings for lead, I noticed that the addition of an excess of potassium iodide to a lead salt caused the precipitated iodide to change from an orange-yellow to a white colour. This change I found was due to the formation of a double iodide of potassium and lead. Although discovered nearly a year ago it is only recently that I have had time to investigate the compound fully. The result I am tempted to lay before the chemical world, chiefly on account of the remarkable property the iodide possesses of being decomposed by water, with the formation of iodide of lead (PbI_2) and potassium iodide; thus:— $PbI_2 \cdot 2KI = PbI_2 + 2KI$. The action of water appears to be catalytic, unless, indeed, the hypothesis be advanced that potassium iodide forms a hydrate which is decomposed when its solution is evaporated; in which case:—



This peculiar property makes the iodide a most delicate test for water. I have often detected the presence of moisture in chloroform and ether when anhydrous copper sulphate failed to show the least change of colour.

Preparation.—One grm. of lead nitrate is dissolved in 10 c.c. of distilled water. A saturated solution of potassium iodide is then added until the precipitate is just dissolved. The solution is allowed to stand for a few minutes, when a copious crystalline precipitate of the double iodide falls. The supernatant liquor is quickly poured off (and preserved), and the precipitate shaken for a few minutes with about 10 c.c. of absolute alcohol. This removes any excess of KI from the double iodide, the latter being insoluble in alcohol. The alcohol is then poured off, and the salt quickly dried with filtering paper. It should be preserved in a stoppered bottle over calcium chloride.

The salt occurs in silky acicular crystals of an almost white colour. So easily is it affected by moisture that, when exposed to the air of a room for a few minutes, it begins to decompose. This is evidenced by it losing its almost white colour and becoming distinctly yellow. When gently heated it also becomes yellow, but it resumes its original colour on cooling. Heated more strongly it is partially decomposed, iodine vapours appearing.

The salt is very slightly soluble in boiling chloroform, but is easily dissolved by a strong solution of iodide of potassium. The mother-liquor from which the double iodide has been obtained holds a certain further amount in solution. This can be recovered by adding 1 c.c. or less of lead nitrate solution (10 per cent), stirring until the PbI_2 is dissolved, and allowing the precipitate to form. The solution can then be decanted from the precipitate and treated again as before, until the iodide is all precipitated and the excess of potassium iodide exhausted.

When the saturated solution is left for twenty-four hours in a cool place there is sometimes a considerable deposit of iodide, which then takes the form of very fine acicular crystals of great length (15 m.m.).

It is worthy of notice that, while pure water decomposes the compound, a strong solution of potassium iodide dissolves it.

Composition.—The mean of three analyses gave—

	Calculated to $PbI_2 \cdot 2KI$.	Found.
PbI	57.963	57.855
KI	42.037	42.145
	100.000	100.000

(The KI was estimated by difference).

The composition is therefore $PbI_2 \cdot 2KI$.

Charters Towers Mining Institute,
Charters Towers,
Queensland, Australia.

ON THE DECOMPOSITION OF CONCENTRATED SULPHURIC ACID BY MERCURY AT ORDINARY TEMPERATURES.

By CHARLES BASKERVILLE and F. W. MILLER.

In a recent number of the *Journal of the American Chemical Society* (xx., p. 100) Mr. J. R. Pitman denied the statement of the authors that concentrated sulphuric acid is decomposed by mercury at the ordinary atmospheric temperature, 20° C. Mr. Pitman leaves himself open to the same criticism that could with justice be urged against us, namely, failure to report the *strength* of the acid obtained in any other way than that by specific gravity.

As is well known, sulphuric acid 1.84 sp. gr. may contain either 95.6 or 99.2 per cent H_2SO_4 . The apparatus at our command does not permit a determination of the specific gravity more accurately than the second decimal place. Sulphuric acid sp. gr. 1.839 contains 99.70 per cent H_2SO_4 . The acid used by us in the experiments reported (*vide supra*) contained 99.65 per cent H_2SO_4 by analysis. Acid of that strength placed in an air-tight glass-stoppered bottle with pure mercury gave off in twenty-four hours sufficient sulphur dioxide to bleach a very dilute potassium permanganate solution. In several instances, after allowing a longer exposure, the gas was easily recognised by its odour when the stopper was removed.

Whether or no any reaction occurs depends entirely upon the *strength* of the acid used, as independently noted later by M. Berthelot, who states that "boiled sulphuric acid has a slow action on mercury, forming sulphate and giving off sulphurous acid." He also states that the reaction occurs only at the maximum concentration (*CHEMICAL NEWS*, lxxvi., 325; *Comptes Rendus*, cxv., No. 20). When our paper was read before the Society in Detroit, Dr. E. W. Morley, in the discussion following, stated that he had previously made similar observations, but had not published them. In a recent letter from him he stated that, when freshly distilled cold sulphuric acid was in contact with mercury, he found it impossible to maintain a vacuum on account of the evolution of from 1 to 2 c.c. sulphur dioxide per day.

An ordinary hydrometer may indicate 1.84 acid, when in fact it may be 1.837 sp. gr., or 94.2 per cent acid, which fact we have observed. We have lately (since the appearance of Mr. Pitman's paper) observed that 95 per cent acid does not seem to react in the cold with mercury. Further, we have seen that during three weeks in this climate 95 per cent acid dropped to 94.2 per cent by allowing the bottle to remain open a few minutes each day. The content of acid more or less exposed to the moisture of the air while being used in nitrometers could easily change—the stronger the acid the more rapid the change. Acid of 94 per cent serves very well for desiccation in ordinary gas analysis, and has no apparent action upon the mercury; however, the authors insist that

concentrated sulphuric acid of 99.5 per cent strength is decomposed by mercury.

University of North Carolina.

METALLURGICAL NOTES.

WILBORGH'S THERMOPHONES.—FERROSODIUM.— MANGANESE BRONZE.

By SERGIUS KERN, M.E., St. Petersburg.

1. THE very handy thermophones of Prof. Wiborgh, of Stockholm, are in constant use at the Baltic Works for several purposes:—For measuring the heat in chimney flues; under the boilers; for taking the temperature of copper alloys before casting; for the same purpose, in connection with the small Bessemer for steel castings.

The thermophones, as many perhaps know, consist of cylinders of fire-resisting material, containing inside a fulminating compound, and represent a simple method for approximative estimation of heats in metallurgical operations, quite good enough for ordinary purposes.

For instance, the cylinder is thrown into a ladle of cast-iron ready to be poured into the converter; long before the ladle is brought up to the mouth of the vessel, the cylinder explodes. The time in seconds is noted, and in the tables attached to every box (50 thermophones) the temperature may be read. Sometimes, through a fault, the cylinders do not explode. We make another trial. In some instances we throw two thermophones at once, and take the mean number of seconds elapsed before the explosion.

We have used these thermophones for more than a year at the above-named works in the steel foundry, and have certainly found a great advantage in knowing the temperature of cast-iron run out of the cupola for the small Bessemer; next we know, at intervals and at the end, the temperature of the liquid metal in the converter itself: we turn the vessel for a few seconds, and measure the heat by throwing in a cylinder.

By such means we easily know what must be done with the metal, and for what castings, big or small, the steel is good.

The average temperature of cast-iron before pouring into the converter is 1400° to 1500° C. But in cold runs of the cupola the temperature comes down to 1000°. When ready the steel is at 2200° to 2400°, and often higher: these higher temperatures cannot be estimated by the described method.

We have ascertained the temperature of crucible steel prepared in our naphtha-fired furnaces; the temperature just before pouring, for steel castings also, is 2200°. Such experiments prove that naphtha-burning furnaces for melting steel are far more convenient in comparison with the coke furnaces, and nearly equal to the Siemens gas-crucible furnace; but are more cheap and handy than the latter when small quantities of steel are melted at a time—say 3 cwts. Morgan's crucibles are used by us; they stand well several heats. But, strange to say, there is not a single works here making crucibles for the market. Several big Russian steel establishments make crucibles, but for their own use only.

2. I had an opportunity to experiment myself, and to witness some others try the ferrosodium supplied by Mr. Blackwell, of Liverpool. In crucible steel this reducing flux makes the slags thinner, but aluminium had still to be used. But in casting a steam cylinder jacket, some 4 tons in weight, there was a visible improvement. The charge in the cupola contained 75 per cent of machine scrap and 25 per cent of pig-iron. The ferrosodium was thrown into the ladle, and gave, when the cast-iron was poured into it, dense fumes. The regulations, here, prescribe 2000 English lbs. as breaking load for test-pieces (1 inch x 1 inch and 12 inches long); the cast-iron

treated with the ferrosodium gave, as breaking-strain, 2200 and 2800 lbs. The alloy cleans the metal in the ladle. A firm in the south of Russia gives me the same account.

3. I am working with the assistance of the laboratory of the Obouchoff Works, near St. Petersburg, on bronze alloys, produced by the addition of Mr. Keiffenheim's "special manganese alloy." Properly brasses are made, but they have the qualities of bronzes, without using expensive tin in the charge. The preliminary experiments gave the following results:—

Breaking strain.	Elongation (per cent).
26.4 tons per sq. inch.	24.9 in 8 inch.
22 " " "	15.4 " "

The second test showed the metal imperfectly mixed. A description of further experiments shall follow.

St. Petersburg, New Admiralty.

NOTE ON SOME REMARKABLE THERMO-EFFECTS PRODUCED UNDER THE INFLUENCE OF HIGH PRESSURE.

By H. N. WARREN, Principal, Liverpool Research Laboratory.

THE apparatus made use of, in order to conduct these investigations, consisted in a small platinum tube 1/16th of an inch in diameter by 1/2 inch thick, this being enclosed in a further tube constructed of cold-drawn steel, 1 1/2 inches in diameter and 2 feet long, an interior perforation having been made in it presenting a diameter of 1/4 an inch and extending to a depth of 6 inches. The steel jacket itself was furnished with a specially cut thread of 3 inches deep, and mounted with a formidable steel cap, corresponding in thickness and diameter to the bottom of the steel tube, and in length measuring from the extremity of the central excavation, whilst the cap real was also drilled through the top, in order to allow of the insertion of a suitable lever when desirous of examining the internal contents.

In the first experiment 1 inch of the platinum tube was filled with distilled water, the further end being hermetically sealed by means of the oxygen blowpipe, and tightly packed with fine magnesia previous to confining it by screwing on the steel cap. The jacket and its contents were now slowly raised to a full red heat and maintained for three hours at that temperature; then taken from the furnace and allowed to cool, the cap unscrewed, and the platinum point removed, the quantity of water recovered being about three-fourths of the original quantity inserted. The most curious observation of this remarkable experiment was the powerful odour of ozone perceptible, as well as the presence of small quantities of hydrogen peroxide, both of which the remaining aqueous portion proved upon examination to be impregnated at the same time with substances one would least have expected to find under such circumstances.

In the second series of experiments the temperature was urged to whiteness; upon examination being made, the entire contents of the platinum tube had disappeared, leaving the platinum surface comparatively unaffected, whilst that portion of the magnesia lining in closest proximity to the platinum had become somewhat crystalline.

In the third series of experiments 5 grains of cane-sugar were enclosed, and treated at a red heat for five hours, the contents upon examination amounting to a few drops of water, accompanied by a powerful odour of acetylene, as well as the production of an extremely hard and crystalline graphite, presenting when microscopically examined an appearance closely resembling the best cut

retort carbon, with a corresponding brilliancy to that of crystalline silicon.

In the last series of experiments, the author being anxious to ascertain the electrothermic results obtainable by the enormous pressure thus exerted, the steel cap of the apparatus had accordingly to be somewhat altered, in order to insulate an inserted platinum wire; accordingly, a perforation having been made through the cap of the apparatus to allow of this being done, 3 c.c. of distilled water were next introduced into the steel jacket itself, and a platinum wire of No. 10 gauge inserted through the small opening, insulated by means of asbestos fibre, and luted as well as possible to the steel. The entire apparatus was now raised again to a dull red heat, whereupon—the projection wire having been brought within close proximity to the steel case—vivid sparks, 1/16th of an inch in length, formed a continuous discharge, which gradually increased in length, but unfortunately came to an abrupt termination by the lifting of the sealed joint, which naturally presents insurmountable difficulties in order to obtain anything like a true closure; further experiments are, however, being contemplated which it is hoped may bring to the front some important changes both in chemical and electrical matters; but the terribly dangerous nature of the experiments, and consequently the great care required when conducting the same, to a great extent retards, in many instances, what otherwise might blossom into valuable results.

Liverpool Research Laboratory,
18, Albion Street, Everton, Liverpool.

ON NEODYMIUM.

By O. BOUDOUARD.

In the year 1885 M. Auer von Welsbach described a method for the separation of lanthanum and didymium, and also for the subdivision of didymium into praseo- and neodymium; this method depends on the difference in solubility, in dilute nitric acid, of the double nitrates of didymium and ammonia, and of lanthanum and ammonia.

In following up my researches on the yttric earths, I have always endeavoured to obtain the earths in as perfect a state of purity as possible. To separate these earths from those of the ceric group, which may possibly be present, the best method is to use sulphate of potassium; the earths of the ceric group give a double sulphate, insoluble in a saturated solution of an alkaline sulphate; those of the yttric group give a soluble double sulphate.

Preliminary trials enabled me to isolate a very small quantity of an oxide having an atomic weight of 142.7, of which the salts gave the absorption spectrum of neodymium, such as was described by M. Auer von Welsbach. This oxide was obtained in the following manner:—The yttric oxides, which had already been purified, were converted into sulphates, and the aqueous solution of these sulphates was treated with an excess of sulphate of potassium; this is left for about twenty-four hours, and the double sulphate formed is collected on a filter, then decomposed by hot soda. The oxide, after being completely freed from alkali, is dissolved in nitric acid, and then precipitated by oxalic acid.

I was anxious to see if a fresh experiment would confirm these results. For this purpose 70 grms. of yttric oxides were submitted to the same treatment as just described, and I was thus able to isolate a certain quantity of an oxide of which the solution gave the spectrum of neodymium. After having removed the thorium, by the formation of the double sulphate of neodymium and sodium, insoluble in a saturated solution of sulphate of sodium, I analysed the product finally obtained. The results were as follows:—

Anhydrous sulphate used	..	2.758 grms.
Oxide obtained		1.605 "

This gives as atomic weight for the corresponding metal the figure 143.

The oxide obtained after calcination is of a greenish colour; the oxalate and the anhydrous sulphate have a slightly rose-coloured tint. The crystallised sulphate is rose-coloured, and is less soluble in water than the anhydrous salt; this solubility is more marked in the cold than when the solvent is hot; these properties relative to solubility are characteristic of all the rare earths.

The absorption spectrum of the solution of the sulphate is characterised by the following wave-lengths:—

591.5 to 584	Vague.
584 " 572	Very strong.
523 " 519	Strong.
512 " 508	Faint.
480	Very faint.
470	Very faint.

I was able to compare this spectrum with that of a solution of neodymium belonging to the laboratory, and thus assured myself of their identity; the very faint lines (480 and 470) are very likely due to traces of praseodymium.

The conclusions to be drawn from the above facts are that neodymium gives a double sulphate with potassium, more soluble than that of praseodymium, which enables us to make a more rapid separation than is perhaps possible by fractional crystallisation. I intend applying this method, founded on the relative solubilities of the double alkaline sulphates, to the study of the didymium salts. —*Comptes Rendus*, vol. cxxvi., No. 12.

ON THE YTTRIC EARTHS CONTAINED IN THE MONAZITE SANDS.*

By P. SCHÜTZENBERGER and O. BOUDOUARD.

For the last three years I (O. B.) have been working on the yttria earths. I commenced this work under my esteemed master the late P. Schützenberger, who did not hesitate to collaborate with me, in face of the difficulty of the question (*Comptes Rendus*, cxxii., p. 697; cxxii., p. 782). It is the results which we obtained that form the subject of this memoir.

By *yttric earths* we mean all the earths giving double potassic sulphates soluble in a saturated solution of an alkaline sulphate, but precipitated by oxalic acid and oxalate of ammonia; this last reagent not being employed in excess. The proportion contained in the monazite sand that we had at our disposal was unfortunately small, so that we were obliged to use several kilograms of the mineral in order to obtain a sufficient quantity of the oxides.

Extraction of Yttric Earths.—The mother-liquors of the double sulphates are precipitated hot by potash. After washing with warm water, the oxides are dissolved in nitric acid, and re-precipitated by ammonia. This treatment is repeated several times, so as to make quite certain that all traces of alkali have been eliminated; this precaution is indispensable for certifying to the correctness of the figures obtained after the calcination of the sulphates. Finally, the oxides are dissolved in nitric acid and precipitated by oxalic acid. The oxalate is washed by decantation and filtration, then dried and calcined to obtain the oxide, which exists as a yellow powder easily soluble in mineral acids.

Method of Fractional Crystallisations.—The yttric oxides are dissolved in nitric acid, and the nitrate converted into sulphate. The sulphate is gently calcined so

as to drive off the excess of sulphuric acid; after this dehydration and cooling, it is taken up by cold water. This solution must be heated on a water-bath; we then obtain a crust of rose-coloured crystals, which are collected. When there is a sufficient deposit, the mother-liquor is decanted and a further evaporation started. This must be repeated time after time, and we thus obtain a series of fractional crystallisations which have been successively analysed:—

First Crystallisation.

Water of crystallisation	21.95 per cent
Hydrated sulphate used	4.9884
Anhydrous sulphate obtained..	3.8931
Atomic weight	105.9
Anhydrous sulphate used	1.8848
Oxide obtained	0.9798

Second Crystallisation.

Water of crystallisation	21.92 per cent
Hydrated sulphate used	5.1254
Anhydrous sulphate obtained..	4.0015
Atomic weight	105.75
Anhydrous sulphate used	1.9967
Oxide obtained	1.0374

Third Crystallisation.

Water of crystallisation	21.98 per cent
Hydrated sulphate used	10.4708
Anhydrous sulphate obtained..	8.1739
Atomic weight	104.6
Anhydrous sulphate used	1.7912
Oxide obtained	0.9266

Fourth Crystallisation.

Water of crystallisation	21.71 per cent
Hydrated sulphate used	7.9209
Anhydrous sulphate obtained..	6.1999
Atomic weight	100.6
Anhydrous sulphate used	2.0310
Oxide obtained	1.0347

Fifth Crystallisation.

Atomic weight	100.2
Anhydrous sulphate used	1.4926
Oxide obtained	0.7593

The three first crystallisations (105.9, 105.75, 104.6) were added together and then submitted to a fresh fractionation in four parts. This gave the following results:—

First Crystallisation.

Water of crystallisation	21.80 per cent
Hydrated sulphate used	6.4190
Anhydrous sulphate obtained..	5.0195
Atomic weight	106.3
Anhydrous sulphate used	1.9109
Oxide obtained	0.9949

Second Crystallisation.

Water of crystallisation	21.98 per cent
Hydrated sulphate used	9.9875
Anhydrous sulphate obtained..	7.7907
Atomic weight	105.2
Anhydrous sulphate used	1.7465
Oxide obtained	0.9053

Third Crystallisation.

Water of crystallisation	22.30 per cent
Hydrated sulphate used	2.4220
Anhydrous sulphate obtained..	1.8917
Atomic weight	101.1
Anhydrous sulphate used	1.8897
Oxide obtained	0.9649

Fourth Crystallisation.

Atomic weight	100.1
Anhydrous sulphate used	0.9041
Oxide obtained	0.4598

These two series of results show most conclusively that we have to deal, not with one earth only, but with a mixture of earths. The variations in the figures obtained in the determinations of the atomic weights are a sufficient proof of this.

We shall not in future give more than the series of results which appear to be of the most interest.

The sulphates which gave 106.3 and 105.2 as atomic weights were mixed and submitted to a fresh fractional crystallisation:—

First Crystallisation.

Water of crystallisation	21.79 per cent
Hydrated sulphate used	4.5412
Anhydrous sulphate obtained..	3.5519
Atomic weight	109.25
Anhydrous sulphate used	2.0993
Oxide obtained	1.1067

Second Crystallisation.

Water of crystallisation	21.80 per cent
Hydrated sulphate used	8.2052
Anhydrous sulphate obtained..	6.4519
Atomic weight	108.75
Anhydrous sulphate used	1.9510
Oxide obtained	1.0247

Third Crystallisation.

Water of crystallisation	21.90 per cent
Hydrated sulphate used	10.8672
Anhydrous sulphate obtained..	8.4847
Atomic weight	106.2
Anhydrous sulphate used	2.1320
Oxide obtained	1.1094

Fourth Crystallisation.

Atomic weight	97.3
Anhydrous sulphate used	1.1233
Oxide obtained	0.5648

The two first crystallisations having given 109.25 and 108.75, were mixed and submitted to a further fractionation:—

First Crystallisation.

Water of crystallisation	21.57 per cent
Hydrated sulphate used	8.0642
Anhydrous sulphate obtained..	6.3244
Atomic weight	109.3
Anhydrous sulphate used	2.0611
Oxide obtained	1.0849

Second Crystallisation.

Water of crystallisation	22.01 per cent
Hydrated sulphate used	4.4826
Anhydrous sulphate obtained..	3.4953
Atomic weight	105.0
Anhydrous sulphate used	2.1441
Oxide obtained	1.1107

The sulphate 109.3 was, in its turn, fractionated:—

First Crystallisation.

Water of crystallisation	21.34 per cent
Hydrated sulphate used	4.4474
Anhydrous sulphate obtained..	3.4985
Atomic weight	111.3
Anhydrous sulphate used	1.7942
Oxide obtained	0.9510

Second Crystallisation.

Water of crystallisation	21.97 per cent
Hydrated sulphate used	3.4944
Anhydrous sulphate obtained..	2.7266
Atomic weight	107.2
Anhydrous sulphate used	1.4702
Oxide obtained	0.7680

The sulphate 97.3 obtained by a preceding operation was fractionated again:—

First Crystallisation.

Water of crystallisation	21.83 per cent
Hydrated sulphate used	2.9693
Anhydrous sulphate obtained..	2.3210
Atomic weight	100.7
Anhydrous sulphate used	2.3192
Oxide obtained	1.1821

Second Crystallisation.

Water of crystallisation	21.78 per cent
Hydrated sulphate used	1.5190
Anhydrous sulphate obtained..	1.1881
Atomic weight	93.9
Anhydrous sulphate used	1.1776
Oxide obtained	0.5838

(To be continued).

THE DIGNITY OF ANALYTICAL WORK.*

By C. B. DUDLEY.

(Concluded from p. 187).

PERHAPS I may venture to give you still one more illustration of how, in the course of analytical work, new and apparently hitherto unnoticed reactions may be hit upon, and modifications of methods result. Every chemist who has done much work in determining phosphorus in iron or steel, by the reduction of the molybdic acid of the yellow ammonium phosphomolybdate, and subsequent titration of the reduced solution, cannot fail to have been annoyed by the occasional failure of duplicates to agree. Apparently in the two analyses everything has been done exactly alike, and yet the results do not agree. Every thoughtful chemist cannot fail to have felt at such times that somewhere in the method there were conditions affecting the result that were not fully controlled. During the last six or eight months in our laboratory we have apparently struck one of these hitherto uncontrolled conditions, whose influence is not large, and yet enough to at times cause annoying discrepancies in duplicates, or between different chemists working on the same sample.

In order to make clear what follows it should be stated that, in the ordinary working of this method, the yellow precipitate, after careful washing, is dissolved in ammonia, and this solution then treated with sulphuric acid largely in excess, and diluted to a definite volume, in which condition it is passed through the reductor, and subsequently titrated with standard potassium permanganate. The reductor in common use consists, as is well known, of a tube of heavy glass, about 5/8ths of an inch internal diameter and about a foot long, filled with powdered zinc, the top being fitted with a funnel and the bottom with a stopcock. Below the stopcock a smaller tube carries the rubber cork, by means of which the reductor is fitted to the flask which receives the reduced solution. This smaller tube usually projects into the flask an inch or two, and it is customary to use the pump to draw the liquid through the reductor. This much

being premised, we may say that in a communication from Mr. Porter W. Shimer, one of the members of the Sub-committee on Methods of the Committee on International Standards for the Analysis of Iron and Steel, he, among other things, called attention to the fact that when making a number of determinations on the same sample, all other things being the same, he got a reduced solution that required more permanganate if he prolonged the small tube below the stopcock in the reductor, nearly to the bottom of the flask, than if this small tube projected only an inch or two into the flask. This statement brought afresh to our minds a thought that every one who has worked much with molybdic acid must have had, viz., that reduced molybdic acid is very easily re-oxidised. We accordingly determined to find out, if possible, whether this was actually the case, and, if so, how much this difficulty might amount to. Accordingly, a stock solution of ammonium molybdate dissolved in water was prepared, and a number of aliquot parts of this solution measured out. Now, obviously, there are two chances for the reduced solution to become oxidised by exposure to the air. One of these is from the air in the flask during the reduction, and on the other from the outside air during the titration. Without going into minute detail, it is perhaps sufficient to say that when we reduced an aliquot part of our stock solution, using the short tube of the reductor, and adding the permanganate drop by drop, with continual agitation during the whole titration, we used 22.7 c.c. of our standard permanganate, all figures given being a mean of a number of closely agreeing determinations. When now we made the reduction the same as before, viz., with the short tube of the reductor, but titrated by allowing about 95 per cent of the permanganate required to run into the flask before agitation at all, and finishing the titration drop by drop, we used 23.1 c.c. of permanganate; in other words, so sensitive is a reduced solution of molybdic acid, that it is easy by varying the mode of titration to introduce considerable error. Prolonging now the tube at the bottom of the reductor as suggested by Shimer, which would result, as is apparent, in a diminished exposure of the reduced solution to the air in the flask before titration, we found our aliquot part to use up 23.6 c.c. of permanganate. But even with the prolonged tube there is some exposure of the reduced solution to the air during the reduction. Accordingly, on the suggestion of my principal assistant, Mr. F. N. Pease, we put a measured amount of standard permanganate solution into the flask which was to receive the reduced solution, more than sufficient to react with it, and then prolonged the tube from the reductor, to dip below the surface of this permanganate. Obviously with this arrangement the reduced solution is entirely prevented from air exposure. On making the reduction and titrating the excess of permanganate with standard solution of ferrous sulphate, it was found that the aliquot part had now used up 24.1 c.c. of permanganate,—an extreme difference in amount of permanganate used, under the varying conditions described, of nearly 6 per cent. Obviously, if two chemists were working on the same sample of molybdic acid, one employing the manipulation first described, and the other that last described, the discrepancy between them would be serious. The discrepancy on phosphorus in steel, while the same in percentage, is very much smaller in actual figures, but still enough to be annoying. The work above referred to is not yet quite finished, but enough has already been done to demonstrate that the ordinary method of determining phosphorus in steel can be advantageously modified in the interests of greater accuracy; and also, although not yet rigorously demonstrated, there are strong indications that molybdic acid (MoO_3) is always reduced by zinc to Mo_2O_3 , and that the more complex formulæ, $\text{Mo}_{12}\text{O}_{19}$, $\text{Mo}_{24}\text{O}_{37}$, &c., so commonly given as representing this reduction, simply mean that the conditions under which these formulæ were obtained permitted the re-oxidation of the reduced solution to the extent indicated.

* Presidential Address delivered at the Washington Meeting of the American Chemical Society, December 29, 1897. From the *Journal of the American Chemical Society*, vol. xx., No. 2.

There is another phase of this question we are discussing, "The Dignity of Analytical Work," which will perhaps bear a few words. It seems to be universally conceded that the brain that plans and guides is worthy of more honour than the hand that executes; the general deserves more than the private soldier; the architect, than the builder; the investigator who plans the work, than the chemist who makes the analyses. Few will object to such a distribution of rewards as this, and certainly no one will claim that a chemist who, machine-like, simply follows directions, without thought or interest in the matter, can fairly claim recognition for anything more, perhaps, than manipulative skill and honesty. But, on the other hand, is it fair to say that such analysts can truly be called analytical chemists? Does not the genuine analytical chemist embody within himself, not only the capacity of brain to wisely plan his method of attack, to conceive which one of the possible reactions in the case it will be best to employ, but also the requisite manipulative skill to carry out the line of action decided upon? To my mind these two things—viz., the brain power necessary to plan the work, together with the continual activity of the brain while the work is going on, and the skilled and trained hand requisite to do the work—are necessarily coexistent at the same time in the good analytical chemist, and woe be that chemist who tries to put them asunder. The analyst whom chance or the exigencies of earning his livelihood have thrown into a situation, where day after day he must, for a time at least, do the same thing over and over again, and who does not, even in this situation, use his brain constantly, does not each time he adds a reagent think what is going on in the beaker, does not each time he washes a precipitate think what he is washing out, does not every time he makes a weight take a genuine interest in the result, and even the hundredth time that he makes the same determination, is not on the look-out for some flaw in the method he is using, or some possible new reaction in connection with it; such an analyst I say will stand a good chance to remain a routine chemist all his life.

On the other hand, what shall we say of those chemists who plan out a line of investigation, and are content not to make the necessary analyses themselves? We are quite well aware that at the present time this is a very common method of making investigations, and we can, of course, understand that pressure of other duties may make it impossible to pursue investigations in any other way. But we cannot regard this state of affairs as, to say the least, anything less than unfortunate. If we may trust our own experience, the time spent in making the analyses required by one line of attack on a stubborn problem is most valuable, in the opportunity which it affords for carrying the problem in mind, and planning out other lines in case the one in hand does not succeed. Moreover, still more valuable is it to make the analyses yourself, in that while doing so you so frequently get suggestions from the work that are the very ones upon which final success depends. I wish there was time to illustrate this point as its importance deserves, but the history of chemistry and your own experience will have to furnish them to you. To our minds it is hard to overestimate the importance, especially to a young investigator, of his doing his own analytical work for himself. If we read rightly, this was the almost universal habit of the old masters of our science, and we greatly fear that those chemists who from choice delegate their analytical work will find, after years of such delegation, that their reward of successful investigations is very small.

A single thought farther. At the present time so much applied chemistry is either based on analytical work, or has analytical work as an almost essential constituent of its existence, that in a paper discussing analytical work a few words may not be amiss on the relations between pure and applied chemistry. Without wishing to touch in the slightest degree on mooted or disputed questions, it may not be unfair to say that while the applied

chemistry does truly, as the name indicates, in the mass of his work, utilise or apply the discoveries of others to useful effect, it does not at all follow that in the field of applied chemistry no discoveries yet remain to be made. It is certainly not too much to say that no thoughtful chemist has ever worked for any length of time in any field of applied chemistry without finding himself surrounded with problems involving new and unknown reactions, with problems—am I not safe in saying—requiring for their solution as good appliances, as deep study, and as keen thought, as any that occupy the minds of the pure chemists. These problems continually force themselves upon him, and his only regret in the matter is that the time at his disposal does not permit him to solve them as fast as they arise. A prominent feature of these applied chemistry problems remains to be mentioned, viz., they generally have immediate useful applications as soon as they are solved. The applied chemist usually makes an excursion into the unknown, because some difficulty has arisen in the course of his regular work, or because some new, more rapid, or more economical method of accomplishing results is desired. He may succeed in finding a new reaction, or in utilising an old one, as the basis of a successful commercial process, or in modifying a manufacturing method in the interests of both economy and speed. But whatever his work, the immediate useful application of the information he secures is both his stimulus and guide. He may not be able, from lack of time, to follow his work up, and find the complete relations of the facts ascertained to the other branches of chemistry, but this is his misfortune rather than his fault, and this condition of affairs, viz., being unable to follow out to completion lines of research one started on, is, if we understand the matter rightly, not characteristic of the applied chemist alone. This much being said, let us ask in what respects the pure chemists resemble or differ from those who work in the field of applied chemistry. They certainly are alike in this, that neither of them can devote his whole time to original work. Both must devote no small portion of their energy to other lines than making investigations. There may have been a time in the history of chemistry when investigators were so fortunately situated that they could devote their whole time and energy to finding out new truth, and giving their results to the world. All honour to such investigators. Moreover, we all know that occasionally an appropriation of funds or an endowment is made for research in some special field. But truly would it not be too much to say that the work of any large percentage of the pure chemists of to-day is the result of such fortunate circumstances? Furthermore, the pure and applied chemists are alike in that, in their original work, both are seeking for the truth, and, if they are successful, both are adding to the sum of human knowledge. They differ, as it seems to me, principally in this:—First, the researches of the applied chemists being largely made in the interests of corporations or manufacturing establishments, the results of these investigations, in many cases, are not at once available to the world, except in so far as they lead to diminished cost of production. Those who have paid for these researches naturally feel that they should be allowed a period of time at least to recoup themselves for their expenditures, and so they protect themselves either by patents or secrecy. But this is only a knowledge of the truth deferred. Sooner or later the results of the investigations of all applied chemists are added to the great body of accumulated chemical knowledge. The pure chemist, on the other hand, at once gives the results of his investigations to the world, and is quite content if the publication of his researches shall bring him, as his reward, a modicum of appreciation from his fellows. Second, in their original work the pure chemists differ from the applied chemists in the ulterior purpose for which the investigation is undertaken. As has already been stated, the applied chemist usually undertakes an investigation, tries to find new truth, with the avowed purpose of at

once utilising this truth as soon as it is found. Not so the pure chemists. The problems which they attack and solve so successfully have no necessary relation to subsequent utility. The truth which they discover, and put on record, may be found to be useful at some time, but its possible immediate utility or non-utility is not taken into consideration by the pure chemist, either in his choice of a subject for investigation or in the prosecution of his work. The truth for the truth's own sake is his motto and guiding star.

If we have diagnosed the case correctly, then the principal differences between the pure and applied chemist are that the latter withholds the results of his work from the world for a period of time, while the former gives his at once, and that the latter is, in his original work, seeking for truth that is at once useful as soon as it is worked out, while the former neither knows nor cares whether the truth that he discovers is either now or at any future time turned to practical or useful effect. Let me not be misunderstood. I am not attempting to belittle in any sense the work of the pure chemists. They are worthy of all honour and respect. But, on the other hand, I am not at all willing to have the work of the applied chemists made light of or treated as though it were in an inferior field. To my mind there is no occasion for either to belittle the work of the other. The field of chemistry is so broad, the amount of unoccupied ground in every branch of the science is so great, that there is neither time nor energy for struggling as to who is greatest or who is least; but in whatever line a man's tastes, opportunities, or the force of circumstances may lead him, whether as a pure or an applied chemist, whether organic or inorganic, whether theoretical, physical, or agricultural, whether analytical or synthetic, provided in his mind at all times the love of the truth is above all, and honest work is being done, he is worthy of recognition, honour, and respect.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, April 21st, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

Messrs. H. C. Seabrooke, W. W. Cheadle, T. H. Hills, and B. S. Bull, were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Edwin Doward, 30, The Willows, Liverpool; Alexander Grant Russell Foulerton, Dunsdale, Mulgrave Road, Sutton; Oswald Hamilton, Old Stratford, Stony Stratford; George Arthur Jarvis, 66, Millbank, Wellington, Salop; Harry Lancelot Lee, 8, Chichester Street, St. George's Square, S.W.; William Lewins, 43, Exeter Street, Gateshead; Alexander MacGillivray Neilson, Coimbatore, Madras; Henry Trench Waller, Zeehan, Tasmania; Edmund Thomas Whitaker, Swan Hill Court, Shrewsbury; William Ernest Wild, 230, Turton Road, Bromley Cross, near Bolton.

A ballot for the election of Fellows was held, and the following were subsequently declared duly elected Fellows of the Society:—

Henry William Coupe-Annable; Albert Abbott, B.A.; Charles Baskerville, B.S., Ph.D.; Joseph Brierley, B.Sc.; Alfred Campion; Robert Martin Caven, B.Sc.; Frederick Hudson-Cox; Charles Benjamin Dudley, Ph.D.; John Arnold Fleming; Arthur L. Harry Garside, B.Sc.; William Thomas Gidden; Alexander Guthrie, B.Sc.; John Heaton; Lawrence Hislop; Harry Pearson Hodgson; J. Shearson Hyland, Ph.D., M.A.; Edwin Charles Jee, B.Sc.; Samuel Morton Jessop; Edward

Jones, B.Sc.; Thomas Martin Lowry, B.Sc.; George Henry Masson, B.Sc.; Albert Henry Mitchell, B.Sc.; Alfred James Parker; Walter Charles Cross Pakes; Walter Ratcliffe; Francis Pitt Smith; Thomas de Smith, B.A.; Henry Somerville, B.Sc.; William T. Newton Spivey, M.A.; Harry St. John; Samuel Walker, M.A., B.Sc.; John Alexander Williamson; Thomas Barlow Wood, M.A.; Samuel Allinson Woodhead, B.Sc.

Of the following papers those marked * were read:—

*49. "*The Carbohydrates of Barley Straw.*" By C. F. CROSS, E. J. BEVAN, and CLAUDE SMITH.

The authors have continued their investigations of growing barley, extending their observations to the plant under the artificial conditions induced by the removal of the ears at the flowering stage. The diversion of the energies of the plant from its normal seed-bearing function proved to be without influence upon the proportion of furfural-yielding carbohydrates to total carbohydrates. The constancy of this ratio has now been established under the widest possible variations of the conditions of growth, natural and artificial, confirming the conclusions that the furfuroids are assimilated with their special constitutional characters and not formed from hexoses by subsequent processes of oxidation.

By the suppression of the seminal function of the plant, the "maturation" of the straw was virtually arrested, and this is shown by the chemical constants determined in the straw grown up to the harvesting period after removal of the ear, compared with the straw grown under normal conditions. The cellulose and "permanent tissue" are 30 and 10 per cent respectively in excess in the normal straw, and conversely the artificial product is much more readily hydrolysed by acids, and the resulting solutions (after neutralisation) are fermented by yeast to a much greater extent.

DISCUSSION.

Dr. VOELCKER, speaking from the practical and agricultural rather than from the theoretical side, enquired what was the influence of these different constituents—pentoses, hexoses, and the like—in determining the value of cereal straws at the various stages of growth. The feeding value of straw depends on the period of cutting, but the constituents determining this value were not known.

The results of the plan adopted of cutting off the ears and thus preventing formation of grain had some analogy with those obtained in keeping a clover crop closely cut down. There was an increase of root growth, and a consequent enrichment of the soil by accumulation of nitrogen. It was of interest, therefore, to know what happened in the case of cereal straws similarly treated, and in what respects enrichment took place. If the enquiry could be followed up by actual feeding experiments, some knowledge of the utility of those different complex substances at which the authors were working would be attained.

Dr. HORACE BROWN said that in the case of barley plants which had been deprived of their fruitifying organs the increased percentage of permanent tissue and of cellulose was probably due to the accumulation in the straw of the products of assimilation which under natural conditions would have been utilised by the seed. He also expressed a hope that the authors would soon be in a position to give more definite information about the nature of the fermentable carbohydrates which are produced by the acid hydrolysis of these interesting furfuroids.

Mr. CROSS, in reply, said that he hoped to give effect to some of the suggestions in a repetition of the experiments on a more extended scale during the current season.

*50. "*Isomeric Bornylamines.*" By M. O. FORSTER, Ph.D.

It has been observed by Leuckart and Bach (*Ber.*, 1887, xx., 104) that when camphor is heated with ammonium formate at 200°, it is converted into the formyl-derivative of bornylamine; the base from this derivative melted at 159–160°, and had the specific rotatory power $[\alpha]_D =$

-18.6°. By the reduction of camphoroxime with sodium and amyl alcohol, the author has obtained two bases having the empirical formula $C_{20}H_{19}N$; one of these substances melts at 163°, and has the specific rotatory power $[\alpha]_D = +45.5^\circ$, whilst the other melts at 180°, and has $[\alpha]_D = -31.3^\circ$. Having found that the base prepared by the method of Leuckart and Bach is a mixture of these two compounds, the author proposes to retain the name *bormylamine* for the dextro-rotatory base, and as the compounds are not optical antipodes, to term the isomeride *neobormylamine*.

The formyl, acetyl, and benzoyl derivatives, and the hydrochloride, platinichloride, carbamide, phenylcarbamide, and picrate of both bases have been prepared, and are described in the paper.

*51. "Some Derivatives of Benzophenone." By FRANCIS EDWARD MATTHEWS, Ph.D.

Benzophenone in a moist chloroform solution very readily yields a hexachloride, $C_6H_5Cl_6 \cdot C_6H_5$, on saturation with chlorine and exposure to bright light.

Only one modification of this substance appears to be produced in this manner, and all attempts to introduce a further six atoms of chlorine into the molecule proved fruitless. No hexachloride could be obtained from acetophenone. Benzophenone hexachloride crystallises from xylene in brilliant colourless forms melting at 215°. It is a very stable substance, but on heating at a temperature considerably above its melting-point, evolves hydrogen chloride and benzoyl chloride, and yields a heavy yellow oil, from which trichlorobenzophenone was isolated in the crystalline state.

On decomposition with alcoholic soda, benzophenone hexachloride is decomposed in two directions. One interaction results in the elimination of hydrogen chloride and production of the trichlorobenzophenone, which crystallises from acetone on spontaneous evaporation in large hexagonal prisms or plates, and melts sharply at 131°. The second decomposition is represented by the equation $C_6H_5Cl_6 \cdot CO \cdot C_6H_5 + NaOH = C_6H_5Cl_6 + C_6H_5 \cdot CO_2Na$, and the resulting benzene hexachloride is decomposed, producing 1:2:4-trichlorobenzene. The benzoic acid produced in the hydrolysis was identified.

Benzophenonehexachloride on nitration yields a mononitro-derivative of the formula $C_6H_5Cl_6 \cdot CO \cdot C_6H_4(NO_2)$, which crystallises in pale yellow needles and melts at 159°. This compound is decomposed by alcoholic soda in the same way as the parent substance. The products are 1:2:4-trichlorobenzene, metanitrobenzoic acid, a small quantity of an azo-compound, and metanitro-trichlorobenzophenone. The last substance forms pale yellow hexagonal plates from acetone and melts at 143°.

Benzophenone hexachloride dissolves in hot fuming sulphuric acid, producing a sulphonic acid. From this acid the barium salt was prepared; it crystallises in tufts of needles and contains $7\frac{1}{2}$ molecular proportions of water. It is readily decomposed by alkalis, but as the products do not crystallise well they have not been further examined.

*52. "Experiments on Lauronolic Acid." By S. B. SCHRYVER, Ph.D.

An attempt was made to prepare an unsaturated isomeride from the ethylic salt of camphanic acid (oxycamphoric acid) in the same way that tetraconic acid is obtained from terebic acid (Roser, *Ann.*, 1883, ccxx., 255). Ethylic camphanate is, however, unacted upon by sodium ethoxide, the reagent used by Roser to bring about the above-mentioned reaction. Lauronolic acid was then investigated, and a method is described for its preparation on a large scale. It was oxidised with potassium permanganate, but no definite products were obtained, the acid appearing to be totally oxidised, the potassium permanganate acting on it in the same way as on the tetrahydrophthalic acids investigated by v. Baeyer. For this reason, it was thought that lauronolic acid was a hydrogenised benzene derivative. Nitric acid acts on lauronolic acid

violently, yielding principally oxalic acid. In addition, however, it forms *nitrocampholactone*, a neutral product of the formula $C_9H_{13}O_2(NO_2)$, and under certain conditions this substance can be obtained in fairly large quantity. It melts at 171°, is insoluble in water, but soluble in most organic reagents. On reduction with zinc dust and glacial acetic acid, it yields the corresponding *hydroxylaminocampholactone*, $C_9H_{13}O_2(NHOH)$, which melts at 148°, reduces Fehling's solution and ammoniacal silver nitrate in the cold, and is soluble in hot water, from which it separates on cooling in the form of twinned, quadratic prisms. The hydroxylamine derivative, on oxidation with ferric chloride, yields *nitrosocampholactone*—



a bright green compound melting at 117°, and very soluble in most organic solvents. Nitrocampholactone, on reduction with tin and hydrochloric acid, yields *aminocampholactone*, $C_9H_{13}O_2(NH_2)$, a compound readily soluble in water, from which it crystallises in large, pearly plates containing water, which melt at 39°, but effloresce in a vacuum, giving an anhydrous substance melting at 66°. The platinichloride decomposes without fusion above 200°, and is a salt, not of the aminolactone itself, but of the corresponding aminohydroxy-acid—



The nitroso-compound can also be obtained by the action of nitrogen peroxide on lauronolic acid, but was not isolated, as the further action of the peroxide converts it into the nitro-compound, which, indeed, can be more conveniently prepared in this way, as the yield is greater than that obtained by the action of nitric acid. Campholactone also gives a certain amount of nitrocampholactone on treatment with nitric acid.

The hydrochloride of aminocampholactone, on treatment with potassium nitrite, yields *hydroxycampholactone*, m. p. 118°, and a liquid of sweet peppermint-like smell.

If Piloty's recent generalisation (*Ber.*, 1898, xxii., 219), that staple nitroso-derivatives can only be derived from hydroxylamines in which the NHOH group is attached to a tertiary carbon atom, be applicable to cyclic compounds, then neither the Bredt nor the Perkin formula can correctly represent the constitution of camphoric acid.

*53. "The Drying of Ammonia and of Hydrogen Chloride." By H. BRERETON BAKER, M.A.

In a recent paper (*Ann.*, 1898, ccxcix., 267) Gutmann has described experiments on the action of these gases when dried. He comes to the conclusion that the drying of both gases by phosphorus pentoxide is an impossibility, because they are absorbed by the drying agent in a shorter time than is necessary to dry them. The author has repeated the experiments, and finds that this absorption of the gases, when properly purified and dried by lime and sulphuric acid respectively, only takes place when the phosphorus pentoxide is impure. When the oxide contains any quantity of metaphosphoric acid, the absorption takes place as rapidly as, and with similar results to, those described by Gutmann. From the construction of the apparatus, as well as from the similarity of the results, it is concluded that moisture was admitted while the phosphorus pentoxide was present, and that the difference between his results and those of the author published in 1894 is readily explained as due to the production of metaphosphoric acid.

With regard to the vapour density of ammonium chloride, it is pointed out that Gutmann's low results are probably due to his having collected the air, driven out of the Victor Meyer apparatus, over water and sulphuric acid instead of over boiled mercury, which was used by the author. A re-determination of the vapour density of dried ammonium chloride by Dumas's method is published in the paper. When the bulb was protected from the outer air by a phosphorus pentoxide tube terminated by a long capillary, the density of 28.8 was obtained from the dried ammonium chloride, confirming the author's con-

clusion, based on six experiments by Victor Meyer's method, that dried ammonium chloride does not dissociate at 350° .

DISCUSSION.

Mr. SHENSTONE said he believed the precautions taken by Dr. Gutmann for preventing the access of water vapour to the dried ammonium chloride while determining its vapour density were insufficient, as Mr. Baker had shown; he had himself employed the very same precautions for a similar purpose in some of his earlier work on the production of ozone from oxygen, and though the results obtained were independently confirmed, and for some time accepted as correct, yet afterwards it became clear that the precautions in question had not really been sufficient for the purpose. Mr. Baker's explanation of the divergent results obtained by Dr. Gutmann and himself in their studies of the interactions of hydrochloric acid and ammonia, respectively, with phosphoric anhydride, showed the importance of using pure phosphoric anhydride for such work, and he therefore ventured to draw attention to the fact that this substance cannot be prepared by merely re-distilling the crude phosphoric anhydride. Certain precautions must be taken, e.g., it is necessary to employ as low a temperature as is possible in order to avoid volatilising any metaphosphoric acid that may be present, and to seal up the product in glass tubes directly it is made.

The PRESIDENT said, in connection with Mr. Baker's results, it was interesting to remember that, in an elaborate paper in the *Phil. Trans.* for 1885, Professors Ramsay and Young detailed many experiments proving that the statical and dynamical vapour pressures of ammonium chloride were identical, and that the vapour pressures found at about 330° compared with the vapour density of about 15 found by them, give a calculated latent heat which amounts to about 34650 grm. units per molecule, or to about 80 per cent of the theoretical value for complete dissociation. Mr. Baker obtained a normal vapour density at 350° , or about twice that of Ramsay and Young. Assuming the tensions of the vapour to be correct, and the density to be really double that found by Ramsay and Young, then the molecular latent heat would be reduced to about one-half of the value just mentioned. But can it be assumed that the tensions of the perfectly dry salt have been determined, or that they would be identical with the values already recorded? A series of such values with ammonium chloride completely free from water, together with vapour density determinations at lower pressures, would give most valuable results, and as the real interest of the research lay in this direction, he hoped the author would attack the problem.

*54. "Note on some of the Properties of Methylene Di-iodide." By H. G. MADAN, M.A.

Methylene di-iodide, CH_2I_2 , has for many years been used by mineralogists on account of its extremely high density (3.34 times that of water). This is, as usual, accompanied by a very high refractivity for light, which has been fully examined by Dr. Gladstone (*Trans.*, 1891, lix., 293), who found the index of refraction for yellow light (D) to be 1.756 at 10.5° , and the author has obtained nearly identical values with a different specimen. The coefficient of dispersion ($\mu_{\text{H}\gamma} - \mu_{\text{H}\alpha} = 0.062$) is decidedly higher than that of carbon disulphide, and, but for its doubtful permanency in light, methylene di-iodide might advantageously replace the latter substance in spectroscopic prisms, especially as its boiling-point is very high (181°), and as it is practically non-inflammable. A few hours' exposure to strong sunlight turns it a light orange colour, and an exposure of ten days deepens this to a red tint.

At a temperature of 100° sulphur dissolves in methylene di-iodide fairly easily, but some crystallises out on cooling; enough, however, remains in solution at ordinary temperatures to raise the index for D to 1.778.

Methylene di-iodide dissolves phosphorus readily and

abundantly; in fact, its own weight (or, taking into account the relative densities, about twice its volume) of phosphorus may be dissolved in it without reaching the point of saturation at ordinary temperatures. The resulting light yellow liquid has, as might be predicted, a very high refractivity, the index of refraction for the D line being 1.95 at 14° . The third decimal place is not given, owing to a fact not readily explained, which was also noticed by Gladstone and Dale (*Phil. Mag.*, 1859, [iv.], xviii., 30), in dealing with solutions of phosphorus in carbon disulphide; viz., a difficulty in obtaining a sharp image of the spectrometer slit through the solution. The solution is, unfortunately, readily acted on by light, like similar solutions of phosphorus, a deposit of red phosphorus being formed.

This solution is far safer to deal with than the solution of phosphorus in carbon disulphide. A few drops poured on filter-paper may be left freely exposed to the air for hours, and even days, without catching fire, the scarcely inflammable solvent seeming to form a kind of varnish over the phosphorus which protects the latter from rapid oxidation.

DISCUSSION.

Mr. VERNON HARCOURT suggested that the presence of phosphorus in the methylene di-iodide might serve the incidental purpose of preventing the colouration of the liquid when exposed to light.

The PRESIDENT asked if Mr. Madan had observed that the production of free iodine on exposure to light was in any way dependent on the presence of oxygen or traces of water. In the case of carbon disulphide, to which the author had referred as giving a deposit in prisms on exposure to light, this was certainly caused by a secondary action, which involved the presence of water, since perfectly dry carbon disulphide enclosed in sealed tubes, and exposed to sunlight, in presence of a dehydrating agent like sulphuric acid did not form any deposit.

Mr. MADAN, in reply to Mr. Harcourt, said that he was afraid that the phosphorus did not seem to have the hoped-for result. In fact, the traces of iodine liberated appeared (as was noticed long ago by Sir B. C. Brodie, *Trans.*, 1853, v., 289) to hasten the conversion of the phosphorus into the red allotropic form; and the solution, whether in a sealed tube or in an open watch-glass, eventually solidified to an orange or red mass. In reply to the President, he said that the methylene di-iodide had been dried as far as possible by calcium chloride before being placed in the tubes for exposure to light, but he would take an early opportunity of ascertaining the result of a more careful and complete desiccation.

(To be continued).

PHYSICAL SOCIETY.

Ordinary Meeting, April 22nd, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

A PAPER by Prof. T. C. PORTER, on "A Method of Viewing Newton's Rings," was read by Prof. S. F. THOMPSON.

If a parallel beam of light from a rectangular slit falls at oblique incidence upon a plane plate of glass, the first two reflections occur at the upper and lower surfaces of the glass respectively, and give two corresponding images that may be formed on a screen. If now, a second glass plate is added below the first, and parallel to it, at a short distance, four images of the slit appear on the screen. But when the lower plate is brought into contact with the upper one, the reflection from the lower surface of the upper plate follows the same path as that from the upper surface of the lower plate, so that only three images are now to be distinguished. For the two glass plates the author substitutes a "Newton's rings" apparatus, and by the above device for eliminating a set of reflections

he is able to restrict the illumination to the light that comes from the two interior surfaces. As thus observed, the colours of the rings are very brilliant. When the plates are very clean, the darkest area of the "black" spot has a sharply-defined edge, similar to that of the black film of a soap-bubble. By using monochromatic light the various sets of rings may be photographed; they appear as several systems of concentric circles, the systems intersecting one another. This method of illumination by a slit enables Newton's rings to be viewed free from all light except that due to reflections at the bounding surfaces of the air-space between the plates. It reveals to the eye the subordinate interference-systems that coexist with the primary rings, and it demonstrates which of these reflections must be taken into account in the theory of the phenomenon. Moreover, it supplies a means for analysing these systems, and it indicates that the interference of monochromatic light is never complete under these circumstances.

Prof. HERSCHEL said it was rather difficult to follow the arguments of the author without witnessing the phenomena. Much complication was introduced by the successive reflections; it was not clear what became of them. There was no doubt as to the advantage of a narrow slit for the illumination. He thought some of the secondary reflections might be got rid of by using plates that were slightly prismatic.

Prof. THOMPSON had, in his own laboratory, verified the advantages of the author's method of illumination. The result was a very sharply-defined first system of rings. Curves of subordinate interference were easily to be observed by this arrangement.

Prof. BOYS noticed in the photograph of the ring-systems that the independent systems of bands were distorted at the points of intersection. The intersecting curves formed a sort of honeycomb, or hexagonal system, instead of a system of curvilinear quadrilaterals. This distortion reminded him of similar effects observed in the photographs of "ripples."

Mr. EDSEX said he had often noticed similar distortions, but he had always been able satisfactorily to explain them as being the result of imperfect focussing. The author had referred to the fact that a thin film when viewed by reflected light appears black. A phase-change of half a wave-length takes place either on reflection at a rarer or at a denser medium; but there is no information from which to decide between these two alternatives. The truth of the assumption that the phase-change occurs at the denser medium seems to depend, so far as experimental evidence is concerned, upon the observation that in Lloyd's bands the central one is black. To produce the Lloyd's bands only one mirror is used; the bands produced by Fresnel required three mirrors. Wernicke performed an interesting series of experiments, in which white-light reflected for various angles of incidence from a thin sheet of glass was examined spectroscopically. The spectrum was crossed by numerous black bands, and from the position of these bands in the spectrum the thickness of the glass was calculated. The calculated thickness when the angle of incidence was great differed from that obtained with small angles of incidence; the conclusion was that when light is internally reflected, even at an angle of incidence less than the angle of total reflection, a phase-change is produced. If the space between the two plates in Prof. Porter's experiment were filled with a substance of higher refractive index than glass, a confirmation, or otherwise, of this result might be obtained.

Dr. S. P. THOMPSON then exhibited a model apparatus, made by the Helios Company, to illustrate the three-phase method of transmitting power. It consists of a small generator, driven by hand, and a small motor. The generator may be separately excited by a secondary battery; it has three independent coils. The six ends of the coils are connected to six commutator rings. The motor

has three corresponding pairs of opposite coils; these can be grouped in various ways for connection to the brushes of the generator. The six coils are on a hinged frame, so that if necessary they can be laid down flat for other rotation experiments. Two armatures are provided, either of which may be used. The first is an iron wheel with peripheral copper bars, arranged like a squirrel-cage; the other is a simple iron disc, without added conductors.

The PRESIDENT proposed votes of thanks, and the meeting was adjourned until May 13th.

NOTICES OF BOOKS.

The Tutorial Chemistry. Part II. Metals. By G. H. BAILEY, D.Sc. Lond., Ph.D. Heidelberg. Edited by WILLIAM BRIGGS, M.A., F.C.S., F.R.A.S. London: W. B. Clive, University Correspondence College Press. 1897. Pp. 300.

It is assumed that the student has spent at least one year at chemistry before taking up the volume now before us. It is, in fact, more a metallurgical than a chemical work pure and simple, as the manufacturing methods of producing the metals appear to be among its principal features. The first few chapters have to do with atomic weights and the relations between them; the constitution of gaseous and liquid compounds; solution; as well as the nature of chemical reaction, where it is shown that chemical changes are in reality much more complex than they at first sight appear to be. In many cases a careful examination will show that, alongside the main reaction indicated in the equation, secondary reactions are also going on. In chap. viii. we begin with the alkalis, K, Rb, Cs, (Am), Na, Li, their preparation and compounds, followed in the next chapter by the alkaline earths, Ca, Sr, Ba; but we must admit being a little surprised to find in chap. xii. that copper is included with gold, silver, and platinum as a noble metal. Its method of production and manufacture differs *in toto* from those of the other three. The only apparent reason for this seems to be that gold and silver are sometimes found associated with copper. Then in succeeding chapters follow the iron, manganese, chromium, tin, and arsenic groups, all of which are well treated considering the small amount of space available for each. There are three appendices—(1) on crystallisation and crystallography; (2) spectrum analysis; and (3) a list of experiments suitable to each chapter. A good index of eleven pages closes the book.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxvii., No. 12, March 21, 1898.

Reaction of some Reagents on Carbonic Oxide, with Regard to the Analysis of the Air of Towns.—Armand Gautier.—When carbonic oxide is present in the air to the extent of some hundredths, as in the case of the gaseous products of fires and factories, its estimation is simple, by means of absorbing it in acid cuprous chloride. But when it is present only in thousandths or ten-thousandths instead of hundredths, this method—even with M. Saint-Martin's excellent modifications—is of no value. Ammoniacal nitrate of silver cannot be depended on if other reducing agents are present. Permanganate of potash at 1/1000th is reduced very slowly in the cold by carbonic oxide, though at 1/100th its action is more rapid. Iodic acid at 1/100th will not oxidise carbonic

oxide in the cold, but at 170° and 100° the action is very marked. Chloride of gold at 170° is an excellent reagent; with the pure gas the action is almost immediate.

On Neodymium.—O. Boudouard.—(See p. 193).

Some Properties of Phosphorescent Sulphide of Strontium.—J. R. Mourelle.—The author has made mixtures of a sulphide of strontium, having a very strong phosphorescence, with inert bodies, such as sulphate of strontium, barium, or calcium. The results showed that, although the mass became phosphorescent in a uniform manner, the intensity of the light is less when the added sulphate has a low specific gravity than when it has a higher one.

Oxidation of some Amide and Thioamide Compounds.—Echener de Coninck.—The author describes his experiments on the action of the alkaline hypochlorites in the presence of an excess of alkali, on acetamide, sulpho-urea, phenyl-urea, phenyl-sulpho-urea, methylglycocol, and oxymethane.

Chloridised Derivatives of Carbonate of Phenyl.—E. Barral.—The bi-chloridised carbonate of phenyl, $\text{CO}(\text{OC}_6\text{H}_4\text{Cl})_2$, has been obtained; it crystallises in white silky needles which melt at 142°, are insoluble in water, only slightly soluble in the cold in benzene and absolute alcohol, though easily so when boiled.

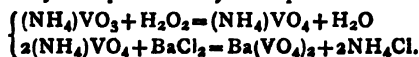
Zeitschrift für Anorganische Chemie,
Vol. xvi., No. 4.

Oxycobaltamines and Anhydroxycobaltamines.—Alb. Mylius.—(1) The author prepares anhydroxycobalt nitrate by adding upon a solution of cobalt carbonate in dilute nitric acid with ammonium nitrate. The raw product is purified by dissolving in hot water containing nitric acid, and, on cooling, the nitrate separates in black needles, which on analysis are found to have the composition $\text{Co}_2\text{O}_2(\text{NH}_3)_{10}(\text{NO}_3)_5$. Anhydroxycobalt chloride is prepared from the nitrate by treating the latter three times with cold concentrated HCl and allowing it to stand for an hour. The green chloride, crystallised from hot water (containing HCl), is obtained in long green needles of composition $\text{Co}_2\text{O}_2(\text{NH}_3)_{10}\text{Cl}_5 + \text{H}_2\text{O}$. Ammonia acts on the nitrate when solutions of the two are shaken up and then concentrated by evaporation. A yellow nitrate separates which has the composition $\text{Co}(\text{NH}_3)_6(\text{NO}_3)_3$. Potassium cyanide acts on the nitrate, with evolution of HCN and NH_3 , forming a cyanide. (2) To prepare chloropentaminocobalt chloride, the freshly-prepared oxycobaltamine nitrate is dissolved in sulphurous acid, and the whole decomposed by strong HCl, when small quantities of the salt are formed. Various oxycobaltamine salts are also prepared.

Quantitative Estimation of Manganese, and the Separation of Iron and Manganese by Electrolysis.—Friedrich Kaepfel.—Manganese can be estimated electrolytically by using a neutral or slightly acid solution, when the manganese is deposited partly as the metal, partly as peroxide. The current must, however, be very strong, and the results are not very satisfactory. The author finds that, by addition of acetone to the solution, much better results are obtained; the precipitate of MnO_2 being obtained as a fine powder. A table of experimental results is given, which agree very well with the calculated ones. The author also attempts the quantitative separation of iron and manganese electrolytically by adding H_3PO_4 to the solution of the sulphates, which succeeds fairly well.

Pervanadic Acid.—Anton Scheuer.—The author prepares barium pervanadate by decomposing a saturated solution of ammonium meta-vanadate in water containing peroxide of hydrogen, by means of barium chloride solution. The barium pervanadate falls as a heavy amorphous yellow precipitate, which must be washed with water containing peroxide until all the ammonium chloride is

removed, and then dried over calcium chloride. The reaction may be represented by the equations—



The $\text{Ba}(\text{VO}_4)_2$ can be analysed by measuring the oxygen split off on the addition of dilute sulphuric acid and slightly warming. The reaction is finished when the red colour of the solution has changed to bright green. The vanadic acid present is titrated against standard permanganate solution. Various other pervanadates are also prepared and analysed.

MEETINGS FOR THE WEEK.

- MONDAY, May 2nd.—Royal Institution, 5. (Annual Meeting). Society of Arts, 8. (Cantor Lectures). "The Electric Locomotive," by Prof. Carus Wilson. Society of Chemical Industry, 8. "Self-intensive Refrigeration of Gases, Liquid Air, and Oxygen," by Dr. W. Hampson.
- TUESDAY, 3rd.—Society of Arts, 8. "Senefelder and the Centenary of Lithography, 1798-1898," by Joseph Pennell. Royal Institution, 3. "The Historical Development of Europe," by Samuel Rawson Gardiner, M.A., D.C.L., LL.D.
- WEDNESDAY, 4th.—Society of Arts, 8. "The Revival of Hand-loom Weaving," by Miss Clive-Bayley.
- THURSDAY, 5th.—Chemical, 8. "The Reaction of the Carbohydrates with Hydrogen Peroxide," by C. F. Cross, E. J. Bevan, and Claud Smith. "The Properties and Relationships of Dihydroxytartaric Acid," Part II., by H. J. H. Fenton, M.A. "The Affinity Constants of certain Hydroxy-acids," by S. Skinner, M.A. "Molecular Weights in Solutions of Permanganates, Perchlorates, and Periodates," by J. Murray Crofts, B.A., B.Sc.
- FRIDAY, 6th.—Royal Institution, 3. "Some Leaders in the Poetic Revival of 1760-1820—Cowper, Burns, and Scott," by The Rev. Canon Ainger, M.A., LL.D.
- FRIDAY, 6th.—Royal Institution, 9. "Living Crystals," by Edward A. Minchin, M.A.
- SATURDAY, 7th.—Royal Institution, 3. "Programme Music" (with Musical Illustrations), by Sir Walter Parratt, Mus. Doc., Master of the Queen's Music.

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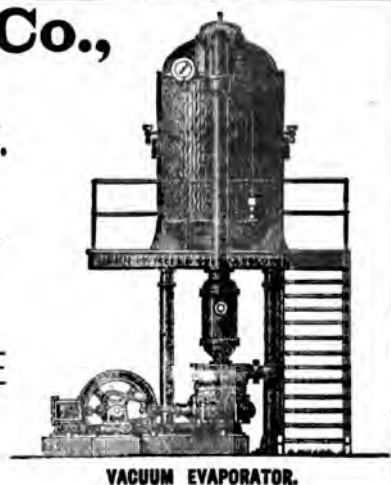
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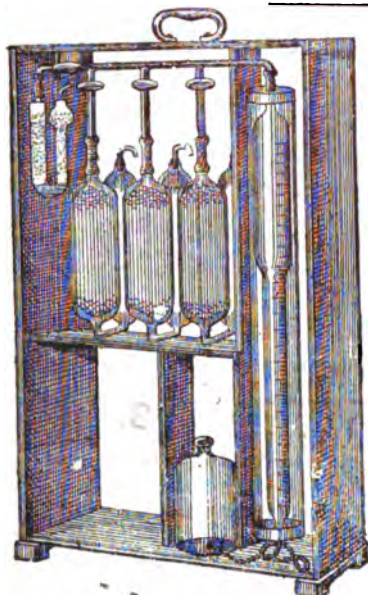
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CONTENTS.

ARTICLES:—	PAGE
Apparatus for Determining the Composition of Ammonia, Sulphur Dioxide, Water, &c., by George George, A.I.C., F.C.S.	203
On an Iodide of Tungsten, by E. Deleqz.	214
On the Yttric Earths contained in the Monazite Sands, by F. Schützenberger and O. Boudouard	204
The Line Spectrum of Silicon, by J. M. Eder and E. Valenta.	206
On the Corrosion of Aluminium, by A. Liversidge, M.A., F.R.S.	207
PROCEEDINGS OF SOCIETIES:—	
CHEMICAL SOCIETY.—The Condensation of Chloral Hydrate with Oxidol.—Note on Hexamethylene and its Derivatives.—The Yellow Colouring Matters of various Adulterants of Sicilian Sunnack.—The Hydrolysis of Starch by Acids.—The Chlorine Derivatives of Pyridine—Lauronic Acid.—The Action of Aluminium Chloride on Camphoric Anhydride—&c.	207
ROYAL INSTITUTION	211
NOTICES OF BOOKS	211
CHEMICAL NOTICES FROM FOREIGN SOURCES	212
MISCELLANEOUS	213
RETININGS FOR THE WEEK	214

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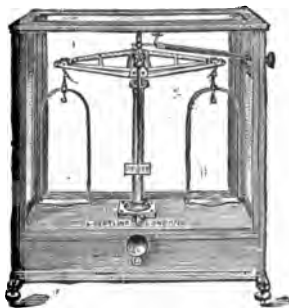
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THE CHEMICAL NEWS.

VOL. LXXVII., No. 2006.

APPARATUS FOR DETERMINING THE COMPOSITION OF AMMONIA, SULPHUR DIOXIDE, WATER, &c.

By GEORGE GEORGE, A.I.C., F.C.S.

THE apparatus shown in the sketch can be used for determining the composition (completely or in part) of a number of gases, and has been found exceedingly useful for lecture-room experiments.

It consists of a bent glass tube, A F, connected by means of a piece of thick-walled rubber tubing to the glass pressure tube, G H. The tube A F is made of Jena glass of about $\frac{1}{4}$ inch bore. The lengths of the limbs of the tube are as follows:—A B and B C each about 3 inches, and C F about 24 inches. A pair of platinum wire electrodes are fused into the tube at L, and a small bulb is blown at K. Before use, the tube is graduated into four parts; the volumes between A and C, C and D, D and E, and E and F being equal. Marks are made at the points C, D, E, and F by means of a file or, preferably, by the use of hydrofluoric acid.

Determination of the Composition of Ammonia Gas.—

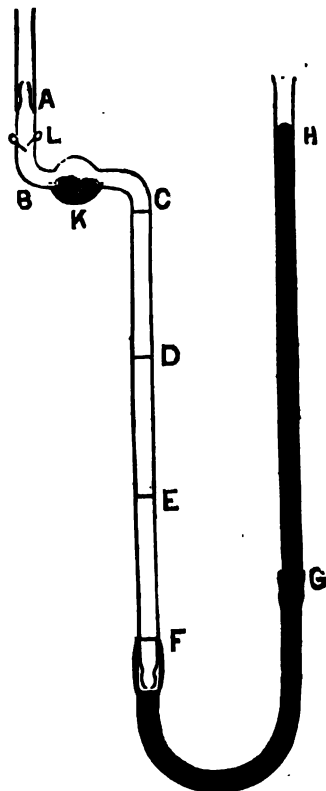
The tube A F, having been graduated and thoroughly cleaned and dried, is attached by means of the rubber tubing to the pressure tube H G. A short piece of rubber is also attached at A; in all cases the rubber should be wired on to the glass tubes. Clean dry mercury is poured into the pressure tube, and the latter is then raised until the whole of the tube A F is filled with mercury. When this is the case, a screw-clip is placed over the rubber tube at A: A F is now ready for filling with dry ammonia. Ammonium hydrate is gently heated and the gas passed through two lime towers to dry it. When it is issuing freely, the delivery-tube is connected with A F by means of the rubber tube at A, and the clip opened. On lowering the pressure-tube, A F is soon filled with the gas, and the clip is then replaced. A short thistle-headed funnel is attached at A, and in it is placed some *fine freshly-ignited* cupric oxide. The pressure tube is raised so that the gas in A F is under pressure, and the clip then opened. The cupric oxide enters the tube, and at the same time some of the ammonia escapes. This prevents any air from entering the tube when the copper oxide is being admitted. Ammonia is allowed to escape until A D is just filled with the gas at atmospheric pressure. By gently tapping the tube, the copper oxide can be made to occupy the bulb K, as shown in the sketch.

The electrodes at L are now connected with a powerful induction coil, and sparks passed. After some time the volume of the gas will almost have been doubled; and when allowed to cool, and the pressure adjusted, the gas will occupy A F (nearly). The bulb K is now heated, first gently and finally to redness. The copper oxide is reduced by the hydrogen, and when all action is over and the apparatus has been allowed to cool down, the residual gas (nitrogen) will be found to occupy A C; that is, two volumes of ammonia gas are composed of three volumes of hydrogen and 1 volume of nitrogen.

To show that sulphur dioxide contains its own volume of oxygen, the tube A F is filled with dry oxygen, and pure sulphur is then admitted. The oxygen is allowed to escape until A D only is occupied by the gas. The portions of tube A B and C D are then heated by means of the Bunsen, and finally the bulb K, which contains the sulphur. When the sulphur takes fire, the flames spread in both directions, and all the oxygen is converted into sul-

phur dioxide. On allowing the tube to cool and adjusting the pressure, the original volume is found to be unchanged. Therefore sulphur dioxide contains its own volume of oxygen. On allowing a strong solution of potassium bichromate to enter at A, all the gas is absorbed.

To prove that water contains two volumes of hydrogen and one volume of oxygen, fill A D with oxygen and then pass in hydrogen until A F is full; that is, there will be two volumes of hydrogen and two volumes of oxygen in the tube. Clamp firmly and spark. On adjusting the pressure, A C will be full of oxygen.



The experiment can then be repeated using one volume of oxygen and three volumes of hydrogen.

The apparatus can also be used for showing that nitrous oxide contains its own volume of nitrogen, whilst nitric oxide contains half its volume of nitrogen. In each case potassium is used to decompose the gas. By using dry air and phosphorus, it can be shown that air consists of a mixture of four volumes of nitrogen to every one volume of oxygen.

This apparatus can be supplied by Messrs. Baird and Tatlock, of London and Glasgow.

Allan Glen's School.
(Glasgow and West of Scotland Technical College).

The Behaviour of some Salts of Platinichlorhydric Acid.—Paul Rohland.—This paper is a continuation of one on the dissociation phenomena of some salts of platinichlorhydric acid with regard to solutions in alcohol and water. It is found that certain salts, as $MgPtCl_6$ and $MnPtCl_6$, crystallise with different numbers of molecules of water at different temperatures, while at temperatures below -3° they separate into their components.—*Zeit. f. Anorganische Chemie*, xvi., No. 4.

ON AN IODIDE OF TUNGSTEN.

By E. DEFAQZ.

VARIOUS reactions have been tried for the preparation of iodide of tungsten. Von Borch tried, without success, the action of the vaporised chloride on iodide of potassium and on iodide of silver, and he had no more success in passing a mixture of the vapour of the same chlorides and hydriodic acid in a tube heated to redness. M. Riche was the first to announce the existence of an iodide of tungsten; Professor (now Sir Henry) Roscoe, in his well-known paper on the halogen compounds of this metal, gave its composition. These two great authorities obtained it by the action of iodine on the metal itself, prepared by the reduction of tungstic acid by hydrogen.

The action of dry hydriodic acid having in several cases given very good results we thought it would be as well to utilise this reaction.

Method of Preparation.—The melted metal, prepared by the use of the electric furnace according to the method adopted by Moissan (*Comptes Rendus*, cxiii., 13), is placed in a boat in the middle of a green glass tube, through which a current of chlorine is kept flowing; it is heated up to a dull red heat, using the precautions recommended by Sir Henry Roscoe. The pure sublimated hexachloride is thus obtained. The chlorine gas is then driven out of the apparatus by means of a current of dry carbonic acid gas, after which a current of dry gaseous hydriodic acid is passed for an hour, while heating that part of the tube nearest to where the hexachloride was first placed to about 400°.

At the commencement of the reaction there is an abundant deposition of iodine, and finally a brown infusible mass is obtained; this must be first washed with pure and dry sulphide of carbon, to get rid of the small quantity of free iodine still present; it is then air-dried and taken up with alcohol at 95°, and finally dried in the oven at 116°. This method requires several precautions: the temperature must not go above 500°, so as to avoid the commencement of the reduction by the hydriodic acid which would otherwise occur, it being preferable to operate on the sublimated hexachloride rather than on the same substance melted.

Analysis.—The iodide is placed in a boat in a glass tube and then heated in a slow current of air; the iodine which is given off by the decomposition is drawn off into a convenient receptacle containing an aqueous solution of sulphurous acid; it is estimated in the form of iodide of silver, and then transformed into the chloride as a matter of verification; the tungstic acid which remains in the boat is weighed; from its weight the quantity of the metal present is deduced.

By this method we found—

	Found.			Calculated for Wl_2 .
W	42.16	42.20	42.24	42.61
I	—	56.82	57.40	57.99
	99.62	99.64	100.00	

Properties.—Iodide of tungsten, Wl_2 , exists in the form of a brown amorphous powder, insoluble in water, sulphide of carbon, or alcohol.

It is infusible, cannot be volatilised without decomposition, giving on contact with the air iodine and tungstic acid. Its density at 18° is 6.9.

Hydrogen is without action until about 500°; above this temperature the iodide is reduced, with loss of iodine.

Chlorine attacks it at about 250°, and gives the corresponding chloride; if the temperature be further increased, compounds containing more chlorine are formed.

Bromine only gives the corresponding bromide at about 350°.

Sulphur and phosphorus give corresponding compounds. Water only acts very slowly in the cold, but more rapidly on boiling; by steaming, the blue oxide is formed.

Carbonic acid has no action until about 500°, and it then gives a brown body, non-volatile and infusible, and which, under the influence of a slight elevation of temperature, burns in the air like tinder, forming tungstic acid.

Gaseous hydriodic acid acts in the same way as does hydrogen at about 500° to 600°.

The aqueous solutions of hydrochloric and hydrofluoric acids only attack it very slowly. Nitric and sulphuric acids and aqua regia decompose it on boiling, and leave a residue of tungstic acid.

Iodide of tungsten is easily attacked by an aqueous solution of potash, and with great vigour, giving off iodine, by melted potash, also by melted alkaline carbonates and mixtures of the nitrates and carbonates.

To sum up, it is very easy to prepare the iodide Wl_2 by the action of dry hydriodic acid on pure hexachloride of tungsten.—*Comptes Rendus*, vol. cxvii., No. 13.

ON THE YTTRIC EARTHS CONTAINED IN THE MONAZITE SANDS.*

By P. SCHÜTZENBERGER and O. BOUDOUARD.

(Concluded from p. 193).

In the whole of the preceding work the process consists essentially of separating as near as possible equal quantities of sulphates, starting from a solution of yttric sulphates, and of analysing the different portions. As the results show, starting with an average atomic weight of 105, we gradually arrive at a series of figures varying from 93.9 to 111.3. In the hope of obtaining better and more rapid results, we changed the method of working. Starting with a solution of yttric sulphates, we crystallised almost the entire quantity, leaving only enough mother-liquor to give on evaporation to dryness 1 or 2 grms. of anhydrous sulphate. The first crystals obtained, after dehydration, were dissolved in cold water, and the solution treated as we have just described. This method has been applied to sulphates which gave on analysis, as atomic weights of the corresponding metals, figures varying from 104.9 to 106.2. Ten operations similar to the one described above were made.

First Residual Mother-liquors.

Atomic weight	98.9
Anhydrous sulphate used ..	1.1065
Oxide obtained	0.5600

Second Residual Mother-liquors.

Atomic weight	99.3
Anhydrous sulphate used ..	2.0725
Oxide obtained	1.0520

Third Residual Mother-liquors.

Atomic weight	101.5
Anhydrous sulphate used ..	1.0925
Oxide obtained	0.5585

Fourth Residual Mother-liquors.

Atomic weight	102.8
Anhydrous sulphate used ..	1.2387
Oxide obtained	0.6365

Fifth Residual Mother-liquors.

Atomic weight	103.3
Anhydrous sulphate used ..	1.2210
Oxide obtained	0.6285

Sixth Residual Mother-liquors.

Atomic weight	101.5
Anhydrous sulphate used ..	0.6210
Oxide obtained	0.3175

* *Bull. Soc. Chim.*, Series 3, vol. xix., No. 6.

Seventh Residual Mother-liquors.

Atomic weight	104.35
Anhydrous sulphate used ..	1.6935
Oxide obtained	0.8763

Eighth Residual Mother-liquors.

Atomic weight	104.5
Anhydrous sulphate used ..	2.0670
Oxide obtained	1.0700

Ninth Residual Mother-liquors.

Atomic weight	106.0
Anhydrous sulphate used ..	1.6529
Oxide obtained	0.8603

Tenth Residual Mother-liquors.

Atomic weight	109.1
Anhydrous sulphate used ..	1.7215
Oxide obtained	0.9053

Finally the analysis of the crystals corresponding to these last mother-liquors gave the following result:—

Atomic weight	114.4
Anhydrous sulphate used ..	1.9173
Oxide obtained	1.0270

B a.—The oxides which gave atomic weights varying from 102.8 to 104.5 were mixed and converted into sulphates. Three successive crystallisations were made:—

B a (1).

Atomic weight	104.2
Anhydrous sulphate used ..	3.2715
Oxide obtained	1.6900

B a (2).

Atomic weight	100.95
Anhydrous sulphate used ..	2.3667
Oxide obtained	1.2072

B a (3).

Atomic weight	95.7
Anhydrous sulphate used ..	0.9084
Oxide obtained	0.4537

A a.—The oxides which gave atomic weights varying from 99.7 to 102.8 were treated in the same manner as B a. The sulphates were crystallised and five portions separated, which gave on analysis the following results:—

A a (1).

Atomic weight	102.15
Anhydrous sulphate used ..	2.3209
Oxide obtained	1.1902

A a (2).

Atomic weight	102.7
Anhydrous sulphate used ..	2.4845
Oxide obtained	1.2773

A a (3).

Atomic weight	101.9
Anhydrous sulphate used ..	2.4267
Oxide obtained	1.2424

A a (4).

Atomic weight	100.3
Anhydrous sulphate used ..	2.3595
Oxide obtained	1.2005

A a (5).

Atomic weight	96.5
Anhydrous sulphate used ..	0.5456
Oxide obtained	0.2736

According to these results we were able to predict the existence of an earth of which the atomic weight of the metal would be equal to 102; the three first deposits

representing nearly the whole of the sulphates used. Admitting this hypothesis, the formula of the hydrated sulphates obtained from all these fractionations would be $(\text{SO}_4)_2\text{N}_2.8\text{H}_2\text{O}$. This formula corresponds to 22.6 per cent of water of crystallisation; experiment has always given figures varying around 22 per cent.

In any case, if we had here to deal with a definite body, we ought, after further fractional crystallisations, to get no sensible variations in the value of the atomic weight found. For this purpose A a (1), A a (2), and A a (3) were mixed and submitted to a new series of crystallisations. The mixture was registered as A a No. 2. The results were as follows:—

A a No. 2 (1).

Atomic weight	103.4
Anhydrous sulphate used ..	3.0900
Oxide obtained	1.5912

A a No. 2 (2).

Atomic weight	101.7
Anhydrous sulphate used ..	2.5080
Oxide obtained	1.2830

A a No. 2 (3).

Atomic weight	98.7
Anhydrous sulphate used ..	0.5350
Oxide obtained	0.2705

The two first crystallisations of A a No. 2 (103.4 and 101.7) were mixed. The oxide obtained by calcining the sulphates at a very bright red heat was dissolved in nitric acid and transformed into sulphate; this sulphate was crystallised, and three portions were obtained, but only the two last were analysed, with the following results:—

A a No. 3 (2).

Atomic weight	100.4
Anhydrous sulphate used ..	0.6158
Oxide obtained	0.3135

A a No. 3 (3).

Atomic weight	96.5
Anhydrous sulphate used ..	0.4170
Oxide obtained	0.2090

The sulphate A a No. 3 (1) was calcined and the oxide converted into sulphate, crystallised, and the mother-liquors only, marked as A a No. 4 (2), were analysed.

A a No. 4 (2).

Atomic weight	98.9
Anhydrous sulphate used ..	0.6726
Oxide obtained	0.3403

A a No. 4 (1) was treated in the same manner as A a No. 3 (1), with the following results:—

A a No. 5 (1).

Atomic weight	103.5
Anhydrous sulphate used ..	1.8865
Oxide obtained	0.9720

A a No. 5 (2).

Atomic weight	99.6
Anhydrous sulphate used ..	1.0248
Oxide obtained	0.5198

A a No. 5 (1) was treated in the same manner as A a No. 3 (1) and A a No. 4 (1), and the mother-liquors only were analysed:—

A a No. 6 (2).

Atomic weight	97.5
Anhydrous sulphate used ..	0.7535
Oxide obtained	0.3791

A a No. 6 (1) was treated in the same manner as A a No. 3 (1), A a No. 4 (1), and A a No. 5 (1), and three

crystallisations were made, which on analysis gave the following results:—

A a No. 7 (1).			
Atomic weight	104.8		
Anhydrous sulphate used ..	2.3485		
Oxide obtained	1.2158		

A a No. 7 (2).			
Atomic weight	98.1		
Anhydrous sulphate used ..	0.4479		
Oxide obtained	0.2259		

A a No. 7 (3).			
Atomic weight	96.05		
Anhydrous sulphate used ..	0.4319		
Oxide obtained	0.2160		

A a No. 7 (1), after undergoing a treatment analogous to what has just been described, gave these results:—

A a No. 8 (1).			
Atomic weight	104.87		
Anhydrous sulphate used ..	3.9965		
Oxide obtained	2.0695		

A a No. 8 (2).			
Atomic weight	99.9		
Anhydrous sulphate used ..	1.2705		
Oxide obtained	0.6455		

Finally, A a No. 8 (1), after a repetition of the same treatment, gave the two following crystallisations:—

A a No. 9 (1).			
Atomic weight	104.65		
Anhydrous sulphate used ..	3.1230		
Oxide obtained	1.6160		

A a No. 9 (2).			
Atomic weight	101.5		
Anhydrous sulphate used ..	1.0405		
Oxide obtained	0.5320		

In this last series, before commencing a fractional crystallisation, the sulphate was always calcined. One might imagine that a calcination at a white heat would facilitate the separation of the earths; as a matter of fact, the sulphates obtained after the solution of the oxide in sulphuric acid showed differences in solubility much more marked, and thus allowed of a much sharper and more rapid fractionation.

According to these several series of results, it is evident that the method of fractional crystallisation is not to be very much depended on for separations; a time arrives when we keep getting a practically constant number during several crystallisations, but at length this figure disappears, to make room for another one. In the hope of getting rid of this cause of uncertainty, we have adopted a method of *fractional fusions*, the results of which will form the subject of our next paper.

THE LINE SPECTRUM OF SILICON.

By J. M. EDER and E. VALENTA.

In a former paper, we have given an account of our investigations on the wave-lengths of the emission spectrum of silicon.

To study this spectrum, we used a spectrograph with one quartz prism, and therefore obtained a spectrum which naturally showed in the less refracted parts a proportionately small dispersion. Since then, we have used a large grating spectrograph, and revised all the results obtained by the former method. We used as standard lines a number of those measured by Rowland, the iron lines measured by Kayser, and the copper lines we had formerly measured ourselves. By the use of these we were able to estimate the wave-length of the silicon lines with the greatest accuracy.

First we satisfied ourselves as to the coincidence of the chief silicon lines in the arc spectrum, as measured by Rowland, with the lines in the spark spectrum. We could not find Rowland's line $\lambda = 2263.507$ in the spark spectrum of silicon, but we confirmed the existence of a line which had already been measured, $\lambda = 2542$ (accurately, $\lambda = 2541.89$), as well as a number of diffused violet and some weak ultra-violet lines. Some very feeble lines of the silicon spectrum could only be obtained with the quartz apparatus and not with the grating—probably on account of the greater amount of light obtainable by the former apparatus.

For investigating the silicon spectrum, we took two alloys—one of silicon and copper and the other of silicon and magnesium. These alloys were used as electrodes—or, equally well, could be used carbon or zinc electrodes—between which, during the passing of an alternating spark, silicon chloride was allowed to drop. By using electrodes of the element silicon, the same results were obtained.

We emphasize once again that the characteristic group of lines, which are defined very sharply, lie in the ultra-violet, so that the identification of silicon spectroscopically is very easy with a quartz spectrograph, but scarcely possible with glass prisms.

The following table contains the results of our measurements related to Rowland's normal line. They extend from $\lambda = 4131$ into the ultra-violet part of the spectrum:—

Wave-length.	Intensity.	
4131.0	4	Diffused.
4128.2	4	
3905.80	3	
3862.75	3	Diffused.
3856.20	3	
3854.00	1	Diffused; indistinct.
3834.4	1	
3826.7	1	
3795.9	2	
3791.1	1	
3191.1	1	
3086.8	1	
2987.77	4	
2881.70	10	Sharp line.
2689.8	1	
2677.4	1	
2673.3	1	
2659.0	1	
2631.39	8	Sharp line.
2568.8	2	
2541.89	8	Sharp line.
2534.7	1	
2533.2	4	
2528.60	8	
2524.21	8	
2519.30	8	Especially characteristic group of lines.
2516.21	10	
2514.42	7	
2506.99	8	
2479.8	1	
2452.22	3	
2446.0	3	
2443.46	2	
2438.86	2	
2435.25	8	Sharp line.
2356.9	1	
2303.3	1	
2219.5	1	
2218.15	1	
2216.76	4	Characteristic group of lines.
2211.8	3	
2210.9	3	
2208.1	3	
2122.8	2	Sharp line.
1929.0	1	

ON THE CORROSION OF ALUMINIUM.

By A. LIVERSIDGE, M.A., F.R.S.,
Professor of Chemistry, University of Sydney, New South Wales.

IN order to ascertain the effects of the weather upon ordinary sheet aluminium, two shallow dishes were made of 1 m.m. or $\frac{1}{16}$ -inch gauge metal, and exposed on the roof of the chemical laboratory of this University from November 23rd, 1893, to December 7th, 1894, or fifty-four weeks.

The metal was made into basins, so as to catch rain-water, and to give the salts, &c., which it might have in solution, a longer time to act upon the metal.

The metal very soon lost its brilliancy, and became somewhat rough and speckled with grey spots mixed with larger light-grey patches; it also became rough to the touch. The grey parts could be seen to distinctly project above the surface, and under the microscope they presented a blistered appearance. This incrustation is held tenaciously and does not wash off, neither is it removed on rubbing with a cloth.

The raised parts are probably due to the formation of a hydrated oxide, but I am leaving the determination of the composition of this until I have a larger quantity at my disposal.

Contrary to my expectations, the cups had not lost weight, but had even increased.

One weighing 13.91 grms. had increased by 0.104 grm., and the other, weighing 13.865 grms., increased by 0.080 grm. After boiling in water for some hours and rubbing, the first still showed an increase of 0.077 grm., and the second of 0.055 grm.

To ascertain the effects of common salt, a plate of the same metal, 3 x 4 inches, and weighing 19.829 grms., was repeatedly dipped in a solution of sodium chloride, and allowed to dry; the alternate dipping and drying was repeated almost daily for three months; the plate lost 0.019 grm., and after washing and rubbing dry 0.059 grm.

My reason for making these experiments is that Mr. H. C. Russell, C.M.G., F.R.S., the Government Astronomer, some years ago tried aluminium cups for a rain-gauge, but found that they were so quickly corroded through that he had to relinquish the use of the metal. If they had been gilt they might, however, have answered well enough. Then, too, it is a very common thing to see aluminium recommended for certain architectural work on account of its lightness and its assumed permanent lustre, this assumption being due to the statements, repeated from book to book, that aluminium is unaltered by exposure to the air, that it is unacted upon by water, hydrogen sulphide, and only slightly by dilute acids—even in modern special treatises such as "Aluminium," by Joseph W. Richards, M.A., London, 1890.

The absolutely pure metal may be permanent in the air, but the best aluminium ordinarily obtainable is in that respect very little, if at all, superior to zinc.

Recently it has been found that sea-water acts upon aluminium with some quickness; hence it is not so perfect a material for torpedo and other boats as was previously thought.

Hence the prevalent idea that aluminium is a metal resembling gold or silver in the property of not oxidising must be relinquished.—*Proceedings of the Australasian Association for the Advancement of Science*, 1895.

Messrs. Perken, Son, and Rayment announce that they have recently perfected a small camera for use in the hand or on a tripod stand; they call it the "Optimus" Ubique improved hand camera. It is suitable for snapshots, landscapes, and portraits, and is fitted with a rising front and also with a horizontal and vertical swing-back. It is a convenient little instrument, and should go well in the coming "shooting" season, especially as the price is not high.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, April 21st, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

(Concluded from p. 199.)

55. "The Condensation of Chloral Hydrate with Orcinol." By J. T. HEWITT, M.A., D.Sc., and F. DIXON, B.Sc.

The authors have re-investigated the substance obtained by Michael and J. P. Ryder by the condensation of chloral hydrate and orcinol. The formula assigned to the substance by Michael and Ryder was $C[C_6H_2(CH_2)(OH)_2]_2CH(OH)_2$. The authors find that on heating the aldehyd and phenol in aqueous solution at 100° for sixteen hours a colourless product is obtained, which melts at 252–253°, and has the formula $C_{16}H_{16}O_6$. The molecular weight, determined by raising the boiling-point of an alcoholic solution, was found to lie between 296 and 336. On heating in an atmosphere of coal-gas, the acid loses a molecule of water and is converted into an almost colourless lactone having the formula $C_{16}H_{14}O_5$. A triacetyl-derivative of the lactone is formed by heating the acid with excess of acetic anhydride. This derivative is colourless, and melts at 189°. On boiling the acid with excess of benzoyl chloride and pouring the hot solution into light petroleum, a tribenzoyl derivative of the lactone is obtained. The tribenzoate has the composition $C_{39}H_{26}O_8$, has no proper melting-point, is apparently insoluble in water, alcohol, and light petroleum, and when allowed to remain in dilute alcohol for some time, is converted into a dibenzoate of the acid which melts at 204°, is readily soluble in alcohol, and has the formula $C_{30}H_{24}O_8$.

56. "Note on Hexamethylene and its Derivatives." By EMILY C. FORTEY, B.Sc.

In a preliminary note on hexanaphthene and its derivatives (*Proc.*, 1897, xiii., 161) it was stated that this hydrocarbon had been obtained from American light petroleum in a state of partial purity, and its identity with hexamethylene was pointed out. It has since been obtained by means of fractional distillation from Galician petroleum. The purest fraction boils almost constantly at 81.75°, and has a specific gravity of 0.7899 at 0°/0°. The following derivatives have been prepared:—*Monochlorhexamethylene*, $C_6H_{11}Cl$, was obtained by passing a current of dry chlorine into the liquid for some hours. It is a colourless liquid boiling at 141.3–141.6° under a pressure of 768 m.m., and has a specific gravity of 0.9991 at 0°/0°. *Dichlorhexamethylene*, $C_6H_{10}Cl_2$, was obtained together with mono- and a little tri-chlorhexamethylene by passing a rapid current of chlorine into heated hexamethylene. It is a colourless liquid when pure, but rapidly turns blue on exposure to air. It boils with slight decomposition at 194–195° under atmospheric pressure. Both mono- and di-chlorhexamethylene give a deep red colouration on warming with concentrated sulphuric acid. On heating dichlorhexamethylene with quinoline, a small quantity of a substance boiling between 80° and 90° is obtained, which appears to be dihydrobenzene. Its alcoholic solution gives a blue colouration with sulphuric acid (Baeyer, *Ber.*, 1893, xxvi., 230). Tetrahydrobenzene was obtained by heating monochlorhexamethylene with quinoline for seven or eight hours. It is a colourless liquid having the properties described by Baeyer (*l.c.*), and boils at 82.3° under a pressure of 764 m.m. *Monobromhexamethylene* could not be obtained by direct bromination of hexamethylene either with or without the aid of a bromine carrier. A small quantity was, however, prepared by the action of an aqueous solution of hydrobromic acid on tetrahydrobenzene. It is a colourless liquid which boils with slight decomposition at 162–163°. The present work will be continued with the view of preparing further derivatives of hexamethylene.

57. "The Yellow Colouring Matter of the Leaves of *Arctostaphylos uva ursi*." By A. G. PERKIN.

Kawaler (*Fahr.*, 1852, 685) found the leaves to contain, besides gallic acid and arbutin, a glucoside ericolin, $C_{33}H_{56}O_{21}$, which yields on decomposition ericicol, $C_{10}H_{16}O$, and a sugar. More recently, B. Degraffe (*Am. Journ. Pharm.*, 1896, lxxiii., 313) has identified the tannin as gallotannin. There is also present a yellow colouring matter of the composition $C_{15}H_{10}O_7$, crystallising in glistening yellow needles; this forms an acetyl compound, $C_{15}H_8O_7Ac$, melting at 188—190°. On fusion with alkali, phloroglucinol and protocatechuic acid were formed. Though resembling quercetin in these points, it has the property of forming deep green solutions with dilute potassium hydrate. Regarding this reaction as due to impurity, the colouring matter was converted (a) into a sulphate, and (b) into an acetyl-derivative, and the colouring matter regenerated from these, but in each case it was unaltered in its behaviour towards alkaline solutions. Oxidation in alkaline solution did not destroy the green colouration until complete decomposition of the colouring matter had taken place. The presence of ellagic acid has also been detected, and thus besides gallotannin, ellagitannin is also present. Broach leaves contain the same colouring matter.

58. "The Yellow Colouring Matters of various Adulterants of Sicilian Sumach." Part V. By A. G. PERKIN and F. J. WOOD.

This paper describes an investigation of the leaves of those plants which are employed for the adulteration of Sicilian sumach (*Rhus coriaria*). We are indebted for samples of these to Mr. P. Gennadius, Director of Agriculture in Cyprus.

The leaves of *Pistacia lentiscus* (shinia) contain a colouring matter of the formula $C_{15}H_{10}O_8$, forming an acetyl compound, $C_{15}H_8O_8Ac$, crystallising in colourless needles (m. p. 204—206°). As on decomposition this yields gallic acid and phloroglucinol, it is identical with myricetin, the colouring matter of *Rhus coriaria*. Two tannins appear to be present: one dissolves in ethylic acetate, yields gallic acid on decomposition, and is evidently gallotannic acid; the other is insoluble, and forms acetic acid, phloroglucinol, and gallic acid on fusion with alkali. When digested with dilute sulphuric acid, the latter yields a red product resembling the anhydrides of the catechol tannins. The further examination of this tannin is reserved. Shinia leaves contain 11·3 per cent of tannin, and appear to be a useful tanning agent, though not suitable for the same purposes as sumach.

The leaves and stems of *Tamaris gallica* and *T. africana* (bruca) contain the same colouring matter. It has the formula $C_{15}H_{10}O_7$, and forms an acetyl compound (m. p. 169—171°). Decomposition with alkali gave phloroglucinol and protocatechuic acid, and with hydriodic acid quercetin and a molecular proportion of methylic iodide were formed. This substance appears to be a new methyl ether of quercetin, distinguished from rhamnetin and isorhamnetin by its ready solubility in alcohol. So little of it was available for examination that its absolute purity could not be guaranteed. The tannin (8·4 per cent) is evidently a mixture of ellagitannin and gallotannin, for both ellagic and gallic acids were isolated as its decomposition products.

The leaves of the *Ailantus glandulosa* contain quercetin. The tannin present (11·9 per cent) is a mixture of ellagitannin and gallotannin, for both ellagic and gallic acids were isolated. This is a worthless tanning agent; the skin, though stained a deep colour, is practically untanned.

The colouring matter of the leaves of *Ficus carica*, the common fig tree, though resembling quercetin, could not be identified, as from 2 kilos. only 0·08 grm. of the substance was obtained. It is nearly devoid of tannin (analysis gave 1·6 per cent), skin being untanned though stained a dirty olive colour.

Gambusso, the stalks of the *Rhus coriaria*, contain a trace of myricetin and some gallotannic acid.

Broach leaves are employed in South Africa in place of sumach (*R. coriaria*), and are also replacing the Cape sumach (*Colpoon compressum*) (*Trans.*, 1897, lxxi., 1132). They contain 19·9 per cent of a catechol tannin, and they form a valuable tannin agent. The colouring matter, having the formula $C_{15}H_{10}O_7$, forms an acetyl compound, m. p. 188—190°, and on decomposition yields phloroglucinol and protocatechuic acid. It differs from quercetin in its reaction with dilute alkali, forming deep green solutions, and appears to be identical with the colouring matter of *Arctostaphylos uva ursi* (see preceding abstract).

The galls of *Pistacia terebinthus* contain a trace of myricetin, whereas Mangrove catch (*Coriops candolleana*) is devoid of yellow colouring matter.

59. "The Hydrolysis of Starch by Acids." By HAROLD JOHNSON.

It has been generally believed that the hydrolysis of starch by acids is similar in character to that effected by diastase, except that the maltose, which is the final product of the action of diastase, is transformed by acids into dextrose. An examination of the products of acid hydrolysis shows, however, that these are not identical with those of diastase conversion, and that when starch is hydrolysed by dilute acids there is neither production of amyloins (molecular aggregates of maltose and the amylin group) nor of maltose. The nature of the products resulting from the reaction can be shortly described as follows:—

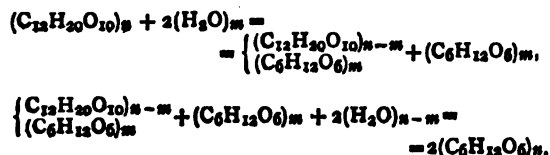
The cupric-reducing powers and the specific rotations of the intermediate substances (fractionated by means of alcohol) as well as those of the total products of conversion, can be expressed exactly in values of dextrose and the amylin group. The relation between the specific rotation and the cupric-reducing power is constant throughout the whole of the reaction, and, given one of these values, the other may be calculated by the equation $[\alpha]_D^{25} 86^\circ = 195 - (195 - 52.8) K_{86}/100$, in which x = the specific rotation and K = the cupric-reducing power in terms of the percentage of dextrose.

The specific rotations of the intermediate substances (separated from the dextrose by precipitation with alcohol) vary between $[\alpha]_D^{25} 3.86$ 80° and 190°. It should be remembered that the intermediate compounds in diastase conversions have rotations which vary from $[\alpha]_D^{25} 3.86$ 190° to 150°, so that the substances from the acid conversions with rotations which fall as low as 80° must differ in character from those obtained by diastase.

The behaviour of the intermediate substances from the acid conversions, when submitted to dialysis, shows that they are definite compounds, and not mixtures of dextrose and other carbohydrates, as, after purification, they dialyse without undergoing a change in specific rotatory power. Moreover, they are unfermentable in the presence of Saaz and Apiculatus yeasts. When treated with phenylhydrazin acetate, they yield gummy precipitates, and on further hydrolysis with acids are completely transformed into dextrose. Their solubility in alcohol decreases as their specific rotation increases.

The products of acid conversion also differ in a marked degree from those of diastase conversion when submitted to the action of diastase. The fall in specific rotation in the case of the products of acid conversion is extremely limited at any time during the reaction. Thus the action of diastase on an acid conversion whose rotation has fallen to $[\alpha]_D^{25} 3.86$ 115° is nil. At 140° the fall is 5° or 6°. At 170° the fall is about 10°, the blue colouration produced by iodine disappearing at this point in the conversion under the influence of diastase. As, however, diastase has a slight degrading action on conversions which give no blue colourations with iodine, and have rotations between 140° and 115°, it is possible that an unreducing dextrin may be produced in the splitting up of the starch molecule; the slight fall in the rotation under the influence of diastase could then be explained by the degradation of this dextrin.

The action of dilute acids on starch can be expressed in its simplest form by the following equations:—

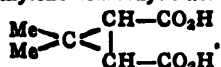


Taking into consideration their properties, the substances intermediate between starch and dextrose may be regarded as molecular aggregates of dextrose and the amylin group $(C_{12}H_{20}O_{10})_n$; the name *gluco-amylin*s will accurately describe them. Gluco-amylin with rotations of about $[\alpha]_D^{20}$ 3.85 80° or 90° have been recognised in commercial glucose under the name of gallisin.

The formation of gluco-amylin by acid hydrolysis is thus explained:—Acids, being able to hydrolyse free maltose, exercise this action also on the maltose in molecular aggregates, and therefore amyloins at the moment of their formation would be transformed into dextrose and gluco-amylin. The molecule of starch is probably formed by the condensation of a large number of molecules of dextrose. In the first place, two molecules of dextrose condense to form maltose, and then a large number of maltose molecules further condense to form starch.

60. "Synthesis of Cis- and Trans-caronic Acid." By W. H. PERKIN, jun., and J. F. THORPE.

The cis- and trans-modifications of caronic acid were obtained by Baeyer (*Ber.*, 1896, xxix., 2796) by oxidising carone with alkaline permanganate, and as these acids when heated with hydrochloric acid yield terebinic acid, Baeyer concluded that they are the cis- and trans-forms of dimethyltrimethylenedicarboxylic acid,—



The authors have succeeded in synthesising these acids in a manner which proves that they have the constitution assigned to them by Baeyer.

When the anhydride of $\beta\beta$ -dimethylglutaric acid is brominated and the product poured into ethylic or methylic alcohol, the following ethereal salts are easily obtained:—*Ethylic- α -bromodimethylglutarate*, $\text{CO}_2\text{Et}\cdot\text{CHBr}\cdot\text{CMe}_2\cdot\text{CH}_2\cdot\text{CO}_2\text{Et}$, is a colourless liquid boiling constantly at 180° (30 m.m.); the corresponding *methylic salt* distills at 155° (22 m.m.). The *acid ethylic salt of α -bromodimethylglutaric acid*, $\text{CO}_2\text{H}\cdot\text{CHBr}\cdot\text{CMe}_2\cdot\text{CH}_2\cdot\text{CO}_2\text{Et}$, is a thick oil distilling without decomposition at 240° (35 m.m.). *Methylic- $\alpha\alpha'$ -dibromodimethylglutarate*, $\text{CO}_2\text{Me}\cdot\text{CHBr}\cdot\text{CMe}_2\cdot\text{CHBr}\cdot\text{CO}_2\text{Me}$, is a colourless somewhat viscous liquid boiling at 172° (22 m.m.). The methylic and ethylic salts of α -bromodimethylglutaric acid and the acid ethereal salt above described, yield, on treatment with alcoholic potash, a mixture of acids which melts at 180–200°, and consists of a mixture of the cis- and trans-modifications of caronic acid. The two modifications may be separated by taking advantage of the difference in solubility of their ammonium salts in hot alcohol, as suggested by Baeyer. The sparingly soluble ammonium salt yields trans-caronic acid, which crystallises from water in lustrous prisms melting at 212°. This acid gives a sparingly soluble crystalline silver salt, but does not form an anhydride. From the readily soluble ammonium salt cis-caronic acid is obtained in plates melting at 174°, and yielding an anhydride melting at 54–56°. There can be no doubt that these acids are identical with the caronic acids prepared by Baeyer.

The authors are investigating the action of the bromo-ethereal salts described above on the sodium derivative of ethylic malonate.

61. "Preparation of Solid Ammonium Cyanate." By JAMES WALKER and JOHN K. WOOD.

In connection with previous experiments on urea-formation (*Trans.*, 1895, lxvii., 746; 1897, lxxi., 489), it was thought desirable to make an attempt to prepare ammonium cyanate in the solid state. Liebig and Wöhler (*Ann. Phys. Chem.*, 1830, [ii.], xx., 393) by passing the vapour of cyanic acid into dry ammonia, obtained a product which they held to be a basic ammonium cyanate. From the description of their experiments, it appears likely that what they actually had in their hands was the normal ammonium cyanate mixed with considerable quantities of urea and of cyanamide. The reason why they did not succeed in preparing the normal cyanate in the pure state is that the heat of combination of the ammonia and the cyanic acid is so great that, under the conditions chosen by them, a large proportion of the ammonium cyanate was decomposed, with formation of the above impurities and of ammonia. If this heating is avoided, it is a matter of no great difficulty to prepare the cyanate in a state which nearly approaches purity. A solution of ammonia in anhydrous ether is gradually added to a solution of cyanic acid in anhydrous ether, both solutions being cooled to –20°. A gelatinous precipitate at once separates, which, after rapid filtration and removal of adhering ether, shrinks to friable masses of a pure white solid. If moisture is rigorously excluded during the operations, and the temperature never allowed to rise to the freezing-point, the product is practically pure ammonium cyanate. When freshly prepared, it dissolves in water without residue, yielding a solution which is neutral to litmus. This proves that the product is free from cyanamide and ammonia, and the only impurity to be feared is urea. The substance effervesces on treatment with nitric acid, and no urea nitrate separates. Silver nitrate solution produces a white precipitate, which is soluble in boiling water and separates out on cooling, thus exhibiting the properties of silver cyanate. Portions of the salt, on being analysed as soon after their preparation as possible, gave the following numbers, the cyanate radicle being estimated as silver cyanate.

	Found.	Theory for ammonium cyanate.
NH ₄	30.0	30.0
CNO.. .. .	70.9	70.0
N	46.8	46.7
C	19.8	20.0
H	7.0	6.7

The cyanate may be kept for a long time in closed tubes without undergoing any considerable alteration. Thus a portion which had remained at the ordinary temperature for fourteen days still contained over 95 per cent of cyanate. On heating in a melting-point capillary, the cyanate contracts visibly at a temperature somewhat above 60°, and melts suddenly at a temperature varying between 76° and 89°, according as the heating is conducted rapidly or not, and according to the tightness with which the material is packed. The fused mass, however, immediately solidifies, and does not again melt till a temperature of 128° is reached—urea melting at 132°. A minute quantity of gas is given off during the first fusion, and a litmus-paper introduced into the upper part of the tube is then turned blue. When the experiment is made on a larger scale, a smell of ammonia is perceptible. Corresponding to this liberation of ammonia, it is found that the transformed solid is not entirely soluble in water, a slight flocculent residue of cyanamide remaining. The first fusion observed when the substance is heated doubtless results from the rapid transformation of cyanate into urea, enough heat being evolved to liquefy the urea produced, which thereafter cools to a temperature below its melting-point. The contraction at first observed probably indicates the temperature at which the transformation proceeds at a perceptible rate. After five hours at 61°, a portion of the cyanate was found to be almost wholly transformed into urea, a small quantity of ammonia and

cyamelide being formed at the same time. Twenty hours' heating at 43° effected the transformation of only 28 per cent of the cyanate. On opening the tube, it was again found that ammonia had been produced, and that the residue was not completely soluble in boiling water. The transformation of the solid cyanate into urea seems, therefore, to be always accompanied by a subsidiary transformation into cyamelide and ammonia.

The investigation is being continued with a view to determining the accurate temperature of transformation of the cyanate, if this is possible, and also to effect the preparation of alkyl ammonium cyanates by similar means.

62. "The Chlorine Derivatives of Pyridine." Part I. By WILLIAM J. SELL, M.A., F.I.C., and F. W. DOORSON, M.A.

The authors have examined the action of phosphorus pentachloride on pyridine, and have isolated the following compounds:—(1) A dichloropyridine, m. p. 87–88°; (2) a trichloropyridine, m. p. 49–50°; (3) a trichloropyridine, m. p. 71–72°; (4) a trichloropyridine, m. p. 67–68°; (5) a tetrachloropyridine, m. p. 90–91°; (6) a tetrachloropyridine, m. p. 21–22°; (7) a tetrachloropyridine, m. p. 74–75°; (8) a pentachloropyridine, m. p. 123–124°.

63. "Simple Experimental Illustration of the Law of Multiples." By A. WENTWORTH JONES, M.A.

Calculated quantities—for example, four times the milligram-molecules—of potassium chlorate and perchlorate are weighed into hard glass tubes provided with corks and delivery tubes, each passing by india-rubber connections through the hole in a beehive-shelf, to near the top of a graduated gas jar full of water in the pneumatic trough. After heating the tubes in the full Bunsen flame for a few minutes, gas ceases to come off, the flames are removed, and the tubes allowed to cool and to withdraw their original content of gas from the receivers through the upturned delivery tubes. The relative volumes collected stand, of course, in the proportion 3 : 4 for equal weights of residue in the tubes. In an experiment before a class, the volumes obtained were 146 and 194 c.c., i. e., in the proportion of 3 : 4 almost exactly. After weighing tubes and residues, and then cleaning and weighing the tubes, the difference or weight of residue was, in each case, 0.30 grm.

64. "Lauronic Acid." By R. W. COLLINSON and W. H. PERKIN, jun.

In connection with the experiments made by one of the authors on isolaunonic acid, they have lately examined lauronic acid with the following results.

Lauronic acid is not reduced by sodium amalgam, but when treated with hydrobromic acid is converted into a hydrobromide, $C_{17}H_{33}O_2Br$, which melts at about 133° with decomposition, and when treated with bromine in chloroform solution, yields a substance which melts at 185° and seems to have the composition $C_{17}H_{33}O_2Br$.

Boiling with dilute nitric acid converts lauronic acid into a substance of the formula $C_{17}H_{33}(NO_2)_2O_2$, which is volatile in steam, and melts at 170°.

When a cold solution of the sodium salt of lauronic acid is oxidised with potassium permanganate, a syrupy acid of the formula $C_{17}H_{33}O_5$ is produced, which is at present under investigation.

Lauronic acid, when warmed with sulphuric acid at 30–40°, is completely decomposed, carbon monoxide being formed together with substances which so far have not been investigated.

65. "The Action of Aluminium Chloride on Camphoric Anhydride." By FREDERIC H. LEES and W. H. PERKIN, jun.

In the course of experiments on isolaunonic acid, on which one of the authors has been engaged for some years, an attempt was made to prepare this acid by a method described by G. Blanc (C. R., 1896, cxxiii., 749–752,

Bull. Soc. Chim., 1896, [iii.], xv., 1191–1199), which consists in adding on a solution of camphoric anhydride in chloroform with aluminium chloride. For a long time the authors failed to obtain anything like the yield of isolaunonic acid obtained in this way by Blanc (40 per cent), and on investigating the cause of this discrepancy, they have isolated the following new substances as products of the reaction. (1) A neutral oil corresponding to about 30–40 per cent of the camphoric anhydride employed: This substance, which the authors propose to call ψ -campholactone, boils constantly at 163–164° (50 m.m.), has the formula $C_{10}H_{14}O_2$, and is isomeric with lauronic acid, isolaunonic acid, and campholactone. It is converted by treatment with potash into the corresponding hydroxy-acid, $C_{10}H_{14}(OH)CO_2H$, a thick syrup which readily loses water, reforming the lactone. When ψ -campholactone is heated with concentrated sulphuric acid at 90°, sulphur dioxide is evolved, and on adding water a crystalline acid is precipitated in quantity; this melts at 126°, and proves to be xylic acid, $C_6H_5Me_2CO_2H$ (1 : 3 : 4).

(2) An acid melting at 255–257°, which gives numbers on analysis agreeing with the formula $C_{11}H_{16}O_3$.

The authors are engaged in the examination of this acid and of ψ -campholactone, as they hope in this way to obtain results having a direct bearing on the constitution of camphoric acid.

66. "On the Action of Bromacetal on the Sodium Derivative of Ethylic Malonate." By W. H. PERKIN, jun., and C. H. G. SPRANKLING.

For some months the authors have been engaged in the examination of this decomposition, and although their experiments are far from complete, they wish to give a short account of the results as yet obtained, in view of a recent paper by Harries (Ber., 1898, xxxi., 37), which deals with substances somewhat similar to those obtained by them.

Bromacetal has little action on the sodium derivative of ethylic malonate at 100°, but when these substances are heated at 140–150° in sealed tubes, interaction takes place readily with the formation of ethylic acetalmalonate.

Ethylic acetalmalonate, $(CO_2Et)_2CH \cdot CH_2 \cdot CH(OEt)_2$, boils constantly at 151–154° (15 m.m.), and on hydrolysis with alcoholic potash yields the corresponding acetal-malonic acid, $(CO_2H)_2CH \cdot CH_2 \cdot CH(OEt)_2$, a colourless syrup, which when heated with water at 190° is decomposed with elimination of carbon dioxide and alcohol, and formation of a substance which is apparently the half aldehyd of succinic acid, $CO_2H \cdot CH_2 \cdot CH_2 \cdot CHO$. This is a colourless syrup, which on long standing deposits crystals, reduces Fehling's solution, combines with phenylhydrazine, and yields succinic acid on oxidation with nitric acid.

The authors are investigating these substances, as well as the action of bromacetal on the sodium derivatives of other ethereal salts.

67. "The Sulphonation of Benzophenone and of Diphenylmethane." By ARTHUR LAPWORTH, D.Sc.

Benzophenone, as it contains the group CO —in direct attachment to the benzene nucleus, should afford a considerable quantity of meta-acid on sulphonation. It has long been supposed that the major portion of the product was the para-disulphonic acid, as Stadel obtained para-hydroxybenzoic acid on fusing it with alkali (Ann., 1878, cxiv., 134). The author has been unable to confirm Stadel's observation; the disulphonic acid he obtained by sulphonation yielded almost pure meta-hydroxybenzoic acid on fusion with alkali, and there is therefore no reason to regard the behaviour of benzophenone on sulphonation as exceptional. During the investigation the following substances were obtained and characterised. Benzophenonedisulphonic chloride, $C_{17}H_8O(SO_2Cl)_2$, crystallises from chloroform in large, anorthic plates melting at 136°, and the form, described by Beckmann, melting at 121.5° could not be obtained. The amide forms needles melting

at 157°, and the *anilide* large crystals melting at 177—178°. The *piperidide* crystallises in monoclinic prisms or plates melting at 168°, and has the curious property of forming a transparent jelly when its alcoholic solution is slowly cooled. A brief study of the sulphonation products of diphenylmethane was made. They appear to be a mixture of disulphonic acids, of which one, doubtless the dipara- acid, yields a *chloride*, $C_{13}H_{10}(SO_2Cl)_2$, melting at 124°, an *anilide* melting at 178°, and a *piperidide* melting at 171—172°. When the sulphonation is carried out by means of chlorosulphonic acid, *diphenylmethaneorthosulphone*, $C_{13}H_{10}SO_2$ is also produced; its constitution follows from the fact that, when oxidised, it yields the benzophenonesulphone described by Stadel, the structure of which has been determined.

68. "The Separation of Optical Isomerides." By FREDERIC STANLEY KIPPING and WILLIAM JACKSON POPE.

In a previous paper ("Racemism and Pseudoracemism," *Trans.*, 1897, lxxi., 989), the authors have dealt with a number of the properties of optically active compounds and their externally compensated isomerides. It would seem, however, that much may be learnt respecting the mutual relationships of these substances by a study of their respective behaviour towards solvents which are themselves optically active. In this preliminary note the authors record the results obtained up to the present in some experiments on the separation of a mixture of equal quantities of enantiomorphously related substances by crystallisation from optically active solvents. The first attempts which the authors made to effect such a separation were unsuccessful; inactive mandelic acid, for instance, crystallised unchanged from an aqueous solution of dextrose. Since, however, inactive mandelic acid is a well-defined racemic compound at ordinary temperatures, it might well be that the tendency to form a solid racemic compound would overcome any tendency towards separation due to the presence in the system of a third enantiomorphous substance; the authors were, therefore, led to attempt the separation of an externally compensated mixture of two substances which do not form a solid racemic compound at the temperature employed. These experiments were successful.

An externally compensated mixture of sodium ammonium dextro- and lævo-tartrate forms a solid racemic compound only above 27°, and when crystallised from water at ordinary temperatures crystals of the dextro- and lævo-isomerides separate in equal quantities side by side, and may be mechanically separated. The authors find, however, that when an optically inactive mixture of sodium ammonium dextro- and lævo-tartrates is dissolved in a concentrated aqueous solution of dextrose, and crystallisation allowed to take place spontaneously at ordinary temperatures (below 27°), the crystalline deposit which first separates contains a very considerable excess of the dextrorotatory salt; when freed from dextrose, first by re-crystallisation from water at ordinary temperatures, and then by fractional precipitation from a warm aqueous solution by alcohol, this first deposit yields a quantity of sodium ammonium tartrate having a high specific dextro-rotation. Eleven samples of salt, each obtained from a different solution, have so far been examined; in only one case the salt separated by the method just described was optically inactive, whilst in all the others it was dextrorotatory. Full quantitative details will be published later, but to afford some idea of the extent to which separation occurs, the authors find that from 175 grms. of a crystallised optically inactive mixture of sodium ammonium dextro- and lævo-tartrates, treated as above in six separate and approximately equal portions, they obtained the following six samples of hydrated active salt:—

1.	2.	3.	4.	5.	6.
Weight in grm.:—					
3.2	3.8	3.0	3.7	4.2	3.5
$[\alpha]_D + 15.50^\circ + 22.07^\circ + 22.97^\circ + 23.30^\circ + 20.65^\circ + 10.56^\circ$					

Landolt's value for the specific rotation of sodium ammonium dextro-tartrate calculated on the hydrated salt is $[\alpha]_D = 23.70^\circ$. The authors have satisfied themselves that all the samples examined in the polarimeter were free from dextrose, and that the racemic acid used in making the salt was optically inactive. The results thus briefly recorded seem to show that enantiomorphously related substances are not equally soluble in presence of a third enantiomorphous substance; whether the difference under these conditions will be sufficient in all cases to render a separation of the two isomerides possible, thus adding another to the few methods by which this may be accomplished, is a matter which is still under investigation.

ROYAL INSTITUTION.

Annual Meeting, Monday, May 2, 1898.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

THE Annual Report of the Committee of Visitors for the year 1897, testifying to the continued prosperity and efficient management of the Institution, was read and adopted. Sixty-six new members were elected in 1897. Sixty-three lectures and nineteen evening discourses were delivered in 1897. The books and pamphlets presented in 1897 amounted to about 260 volumes, making, with 632 volumes (including periodicals bound) purchased by the Managers, a total of 892 volumes added to the Library in the year.

Thanks were voted to the President, Treasurer, and the Honorary Secretary, to the Committees of Managers and Visitors, and to the Professors, for their valuable services to the Institution during the past year.

The following gentlemen were unanimously elected as officers for the ensuing year:—

President—The Duke of Northumberland, K.G.

Treasurer—Sir James Crichton-Browne.

Secretary—Sir Frederick Bramwell, Bart.

Managers—Sir William Crookes, Sir Edward Frankland, The Right Hon. George Joachim Goschen, Donald William Charles Hood, David Edward Hughes, Alfred B. Kempe, Hugh Leonard, Sir William Huggins, Thomas John MacLagan, Ludwig Mond, Alexander Siemens, The Hon. Sir James Stirling, Sir Henry Thompson, Sir Richard Everard Webster, Sir William Henry White.

Visitors—Sir Alexander Richardson Binnie, Sir James Blyth, Bart., Charles Vernon Boys, Edward Dent, James Edmunds, Maures Horner, Edward Kraftmeier, Sir Francis Laking, T. Lambert Mears, Lachlan Mackintosh Rate, John Callander Ross, William James Russell, Sir James Vaughan, James Wimshurst, Alfred Fernandez Yarrow.

NOTICES OF BOOKS.

Commercial Fertilisers and Chemicals. By Dr. GEORGE F. PAYNE, State Chemist. Atlanta: G. W. Harrison. 1897. Pp. 116.

THIS Report refers more especially to the State of Georgia, where the author is State Chemist. It appears that Georgia stands a long way ahead of any other State in the quantity of artificial and chemical manures used, and the manufacture of fertilisers in that State has grown into a business of huge proportions. The two principal kinds here used are a simple acid phosphate, and a complete fertiliser made from acid phosphate, cotton-seed meal, blood, tankage or bone phosphate, nitrate of soda, kainit, and chlorate of potash: 15 to 16 per cent of avail-

able phosphoric acid is now obtained from a well-made acid phosphate from Charleston rock,—a very good proportion.

The number of analyses of fertilisers made during the season 1896-97 was 6015, against 4880 in the season before.

The total number of tons of commercial fertilisers used in the United States during 1896 was over 1,500,000, of which over 1,100,000 tons was used in the South-eastern group of States, among which Georgia stands at the head with a record of 335,617 tons, or more than one-fifth of the whole lot together. A new insecticide is described called "grey mineral ash," which is said to be superior to and safer than Paris green; it consists of certain proportions of sulphate, sulphide, and hydrate of barium, carbon, water, and silica; when mixed with water and applied to the crops, the effect is to give off sulphuretted hydrogen. A number of notes in reply to various enquiries that have been made are given, and the report finishes with a large number of tables of the complete analysis of all the different brands of commercial fertilisers used.

The Freezing-point, Boiling-point, and Conductivity Methods. By HARRY C. JONES. Easton, Pa.: Chemical Publishing Company. 1897. Pp. 64.

It has long been known that the freezing-point of a solution of any substance is lower than that of the solvent used. Raoult tried to generalise it thus:—That "one molecule of any complex substance dissolved in 100 molecules of a liquid, lowers the freezing-point of the liquid by nearly a constant amount, which is 0.62." This has been shown not to hold rigidly." The author then goes on to discuss the variations from this rule, and gives some interesting facts concerning them. The results found with water as a solvent are entirely different to those obtained when benzene, or acetic, formic, or benzoic acid is the medium employed. Compounds like the non-electrolytes give a very nearly constant figure with water, approximately 18.8.

The application of the freezing-point method to the determination of molecular weights in solution is described, using Beckmann's apparatus; after which the same method for the measurement of electrolytic dissociation is considered; an ion lowers the freezing-point of a solvent just as much as a molecule, and, if a molecule dissociates into two ions, the freezing-point of a given amount of a solvent will be lowered just twice as much as if it is not dissociated. Thus it is possible for any given dilution to determine the amount to which an electrolyte is dissociated.

Part II, is on the boiling-point method, and it is stated as a well-known preliminary that "the presence of a foreign non-volatile substance diminishes the vapour-pressure of the solvent," and hence raises the boiling-point; there are thus two quantities to measure. The application of the boiling-point method to the determination of molecular weights in solutions is not so delicate as the freezing-point method, as it is very sensitive to barometric changes: a number of forms of apparatus have been devised by Beckmann, by Hite, and by the present author, but we cannot describe them without the diagrams. The conductivity method is described in Part III., and its application to the method of electrolytic dissociation is also here given. The great drawback to electrolytic methods is, that after a continuous current has passed for a short time, the electrodes become polarised, i.e., covered with gas; several devices have been tried to overcome this, but the simplest is to use an alternating current; several methods of effecting the determination are illustrated, and full details given for carrying out the measurements.

The book is a most interesting one, well and clearly written, and printed in clear type, but—and there is always a but—there is no index.

Organic Chemistry for the Laboratory. By W. A. NOYES, Ph.D. Easton, Pa.: Chemical Publishing Company. 1897. Pp. 257.

A MOST important remark, made by the author in his Preface, is that "a satisfactory working knowledge of organic chemistry cannot be acquired without the ability to use German books." We may add that a successful student must also be able to follow the flood of original work which is being published, not only in German but other Continental languages, besides what is done in England. The author has divided the subject into ten principal headings, but he seems to begin rather abruptly with the idea that the student has already a fairly good knowledge of the subject, though there is nothing in the title to indicate that the book is intended for advanced students only.

There is no necessity to analyse the book closely, as the general outline of the system on which organic chemistry is built up is so well known; suffice it to say that the subject is well and clearly explained, with numerous references to original papers throughout. The Index is very complete, and comprises sixteen pages.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 13, March 28, 1898.

Use of Chloride of Palladium for the Detection of small quantities of Carbonic Oxide in the Air, and on the Conversion of this Gas into Carbonic Acid at the Ordinary Temperature.—MM. Potain and Drouin.—The presence of carbonic oxide in the air, even when in very small quantities, can easily be detected by passing the vitiated air in very fine bubbles through a dilute solution of chloride of palladium. Ten c.c. of a solution containing 1 m.grm. of the chloride, acidulated with two drops of hydrochloric acid, are placed in a special flask, through which the air can be drawn; when the apparatus is started, no matter how small a quantity of carbonic oxide is present, the chloride is decomposed, and the palladium is deposited in a blackish film on the sides of the tube. This deposit points to the presence of a reducing agent. The proportion of the gas present can be estimated by colorimetric means, by comparing the colour of a solution which has been tested with that of a similar solution of known strength which has not been so treated. The authors do not claim great chemical accuracy for this new method, but they think it will be of great service from the hygienic point of view on account of its speed.

On an Iodide of Tungsten.—E. Desfarges.—(See p. 204).

Quinoleic Bases.—Marcel Delépine.—The author has studied some quinoleic bases from the thermochemical point of view, in order to determine the thermic variations which accompany their formation, which, as is well known, is generally by the action of an aldehyd, an acetone, or similar substances able to give rise to these functions, on a primary aromatic amine more or less complex. As we have not much knowledge on the subject the author has at present confined his researches to quinolein, quinaldine, and their tetrahydrides. Experiments were made with quinolein from tar, and also the same prepared synthetically, with tetrahydroxyquinolein, quinaldine or α -methyl-quinolein, and tetrahydroquinaldine; these experiments are fully described and commented on, the general conclusions being that the heat of formation is very small, though this may be accounted for by that absorbed by the

water of formation, and other reasons, such as possible polymerisation, &c.

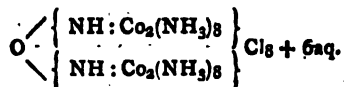
Combination of Organic Bases with various Oxygenated Salts.—D. Tombeck.—It has already been shown that aniline and its derivatives, such as the bases of the pyridic series, are able, like ammonia, to combine with the haloid salts of zinc and cadmium, giving well-crystallised bodies, of which the author has already described the compositions and properties. He now finds that compounds analogous to those used before give corresponding compounds with nickel, cobalt, magnesium, and manganese salts under conditions but slightly different. Aniline again, like ammonia, combines with several oxygenated salts. The general method for the preparation of the compounds formed in this case consists of pouring an excess of aniline into a solution of the metallic oxygenated salt; on agitation the crystalline precipitate is formed.

Zeitschrift für Anorganische Chemie,
Vol. xvi., Nos. 2 and 3.

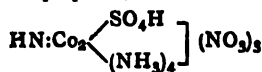
The Combustion of Organic Substances in Aqueous Solution.—I. K. Phelps.—By oxidation with KMnO_4 , the amount of CO_2 obtained from ammonium oxalate, barium formate, and acid potassium tartrate is accurately determined by absorption in standard $\text{Ba}(\text{OH})_2$ solution and titration of the excess of $\text{Ba}(\text{OH})_2$. Chromic acid gives also fairly good results as oxidiser with ammonium oxalate and cane-sugar. The amount of oxygen in some compounds can also be determined by using a known quantity of chromic acid, but the method is cumbersome.

Titration of Sodium Thio-sulphate by Iodic Acid.—Claude F. Walker.—Riegler's method of titrating thio-sulphate with HIO_3 is shown to be very inaccurate, as the end point varies greatly with the volume of the solution, and the nature of the reaction cannot be expressed by any simple chemical equation.

The Constitution of Inorganic Compounds.—Alfred Werner.—Complex cobalt ammonium salts were investigated and carefully analysed. The sulphate had the formula $\text{Co}_2(\text{NH}_3)_8(\text{OH})_2(\text{SO}_4)_2 \cdot 23\text{H}_2\text{O}$ assigned to it. Other salts were also examined, chiefly (1) oxy-di-imido-octamminodicobalt salts, of which the chloride, bromide, sulphate, and nitrate were prepared and examined, and the formula—



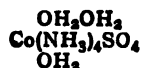
assigned to the chloride. (2) Hydrosulphimide-octamminodicobalt salts, of which the nitrate, sulphate, bromide, iodide, &c., were prepared, and the formula—



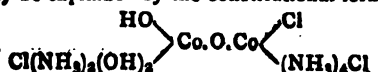
was given to the nitrate.

A New Determination of the Atomic Weight of Nickel.—T. W. Richards and A. S. Cushman.—Already inserted.

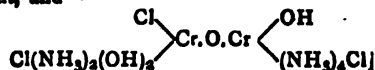
Constitution of Cobalt, Nickel, and Rhodium Bases.—S. M. Jörgensen.—There appears to be a difference in the constitution of complex salts of cobalt and chromium. To investigate this difference the author prepares basic diquotetrammoncobalt sulphate—



chloride, and nitrate, and compares these with the corresponding salts of chromium. He suggests that the difference may be explained by the constitutional formula—



for cobalt, and—

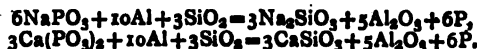


for the corresponding chromium salt.

Electrolysis of Hydrochloric Acid.—F. Haber and S. Grinberg.—The substances formed at the anode and cathode were investigated with acid solution of various strengths, and the following results obtained. Chlorine is evolved at the anode, when the acid is concentrated, with a yield of 100 per cent; and the yield decreases as the acid is diluted. Hypochlorous acid is present in traces in the anode gas when dilute acid is electrolysed. Acid solutions of strengths between normal and $\frac{1}{10}$ th normal give chloric acid, which is accompanied by a small quantity of H_2O_2 . Perchloric acid is present in dilute acid solution. Oxygen is evolved by the electrolysis of dilute acid, and forms about 50 per cent of the anode gas.

Chemiker Zeitung.
1898, No. 25.

Study of Aluminium as a Reducing Agent.—Léon Franck.—Aluminium powder contains small quantities of silica and iron, which must be carefully estimated when aluminium is used as a reducing agent. The author shows that about 0.8 per cent of silica and about 0.34 per cent of iron are present in the purest forms of commercial aluminium powder. He then investigates the reducing properties of aluminium. (1) Phosphorus combines in various proportions with aluminium:— Al_7P_3 , obtained as a greyish black infusible mass; Al_5P_3 as a yellowish grey infusible mass; an unestimated compound forming little dark blue needles; and Al_3P and $2(\text{AlP})$ as balls of metallic appearance. All these compounds are decomposed by water with evolution of PH_3 . (2) Aluminium decomposes phosphates at a high temperature, with formation of phosphorus. By the addition of some silicic acid the decomposition is almost quantitative and according to the equations—



By means of this reaction a new method of estimating phosphorus is described. (3) Aluminium decomposes CO_2 according to the equation $3\text{CO}_2 + 4\text{Al} = 2\text{Al}_2\text{O}_3 + 3\text{C}$; CO according to the equation $3\text{CO} + 2\text{Al} = \text{Al}_2\text{O}_3 + 3\text{C}$; and carbonates according to the equations—



During all these reactions some aluminium carbide is formed. (4) Aluminium is both a good and a sure reducing agent; for it decomposes metallic oxides, forming the metal and Al_2O_3 ; it decomposes sulphates forming sulphur and sulphides, and chlorides forming the metal. (5) A mixture of aluminium powder and sodium peroxide moistened with some water burns spontaneously, with dazzling light.

MISCELLANEOUS.

New York Agricultural Experiment Station, Geneva, N.Y.—The Director of this Experiment Station, Mr. W. H. Jordan, appears to have been doing good and useful work during the past year. Twenty-nine bulletins have been issued during the year on all sorts of subjects connected with or relating to agriculture. These bulletins are widely circulated among the farmers of the State, and carefully prepared abstracts are printed in the news and agricultural papers.

Sanitary Institute.—Sir Joseph Fayrer, Bart., K.C.S.I., M.D., LL.D., F.R.S., has accepted the Presidency of the Seventeenth Congress of the Sanitary Institute, to be held in Birmingham, commencing September 27th.

American Association for the Advancement of Science.—The fiftieth Anniversary Meeting of the American Association for the Advancement of Science will, we are informed, be held in Boston during the week beginning Monday, August 22, 1898. Full particulars can be obtained from Mr. A. Lawrence Rotch, Secretary, Boston, U.S.A.

A Natural Silicic Ether.—E. Drechsel.—By exhausting ordinary white bedding feathers with warm ether, in an extraction apparatus, and leaving the etherised extract to cool, M. Drechsel has obtained a substance, fusible at 52°, easily soluble in chloroform, very slightly soluble in alcohol, and soluble in ether. On analysis it gives figures corresponding to the formula $\text{Si}(\text{OC}_3\text{H}_5\text{O})_4$. This substance may thus be the ortho-silicic ether of a bivalent alcohol, $\text{C}_3\text{H}_6\text{O}_2$, a homologue of cholesteroline. The author has tried to prepare a homologous ether from a solution of chloride of silicon in chloroform and cholesteroline; he obtained a body with analogous properties, melting at 59°. This ether, obtained from feathers, is the first organic compound containing silicic acid met with in nature.—*Centralblatt für Physiol.*, vol. ii., p. 361.

Third International Congress of Applied Chemistry, Vienna, 1898.—1. The third International Congress of Applied Chemistry will be held on the 28th of July, 1898, in Vienna, and will last until August 2nd, inclusive.

2. The subjects of the Congress are as follows:—

- a. Consultations concerning important questions in all departments of applied chemistry, and particularly of those the solution of which is a matter of public interest.
 - b. Agreement upon methods to be considered internationally valid for the analysis of such products as are valued upon the basis of their chemical composition.
 - c. Agreement upon methods to be considered internationally valid for the use of the different chemical industries.
 - d. Discussion on questions of instruction in applied chemistry, and consultations upon general affairs of chemists. And—
 - e. Commencement of a friendly understanding between the representatives of the different departments of applied chemistry at home and abroad.
3. For the settlement of the work of the Congress there will be two general meetings and a further number of special consultations. Besides these there will be excursions for the purpose of visiting Scientific Institutions and Establishments.

4. The special consultations of the Congress will be divided into twelve Sections, as follows:—

I. General analytical chemistry and chemical instruments (general methods of analysis, analytical apparatus, measuring instruments, hydrometers, &c.).

II. Chemistry of food, medical and pharmaceutical chemistry. (The chemical and physical analysis of foods, discussions on chemical questions regarding foods in trades which do not come under the head of any other section; further questions of medical and pharmaceutical chemistry.)

III. Agricultural chemistry. (Analysis of dairy products.)

IV. Sugar industry, starch and grape sugar manufacture.

V. Zymology:—

1. Subsection. Brewing and malting.
2. Subsection. Alcohol and yeast industry.

VI. Oenology.

VII. Chemical industry of inorganic substances. (Sulphuric acid. Soda and chloride of lime manufacture,

alkali industry, artificial manure making, lime and cement industry, industry of illuminants, glass, china and earthenware manufacture).

VIII. Metallurgy and the manufacture of explosives.

IX. Chemical industry of organic substances. (Aniline dyes, dyeing and printing, manufacture of pharmaceutical preparations, chemistry of fats, oils, and lubricants, paper and xylonite industry, tanning, and glue manufacture).

X. Chemistry of the graphic industry. (Photo-chemistry, photographic and chemical printing, colour-printing).

XI. Questions of instruction and the general affairs of chemists.

XII. Electro-chemistry.

Papers to be read at the meeting should be in the hands of the General Secretary, M. F. Strohmayer, Vienna IV/2, Schönburgstrasse 6, not later than the 1st of June. It is requested that no paper be longer than 5 pages 8vo in print.

MEETINGS FOR THE WEEK.

MONDAY, 9th.—Royal Institution, 5. (General Monthly Meeting.) Society of Arts, 8. (Cantor Lectures). "Electric Traction," by Prof. Carus Wilson.

TUESDAY, 10th.—Society of Arts, 4.30. "The Art of William Morris," by F. S. Ellis. Royal Institution, 5. "The Historical Development of Europe," by Samuel Rawson Gardiner, M.A., D.C.L., LL.D.

WEDNESDAY, 11th.—Society of Arts, 8. "Water Gas and its Applications," by Prof. Vivian B. Lewes.

THURSDAY, 12th.—Royal Institution, 5. "Heat," by The Right Hon. Lord Rayleigh, F.R.S., &c.

FRIDAY, 13th.—Royal Institution, 9. "Recent Experiments on certain of the Chemical Elements in relation to Heat," by Prof. W. A. Tilden, D.Sc., F.R.S. Physical, 5. "Galvanometers," by Prof. W. E. Ayton and T. Mather.

SATURDAY, 14th.—Royal Institution, 5. "Programme Music" (with Musical Illustrations), by Sir Walter Parratt, Mus. Doc., Master of the Queen's Music.

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EXAMINATIONS for the FELLOWSHIP (F.I.C.) and ASSOCIATESHIP (A.I.C.) will be held at the Laboratories of the Institute on Tuesday, the 12th day of July, 1898, and three following days.

In the event of 40 Candidates presenting themselves, those for Final Examination will be examined from Tuesday, 19th, to Friday, 22nd July.

Forms of application and an outline of the regulations can be obtained from the Secretary.

Full particulars are given in the "Book of Regulations for Admission to the Membership and Register of the Members and Students," to be obtained on application to Messrs. Blundell, Taylor, and Co., No. 173, Upper Thames Street, London, E.C. Price 2s., post free.

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CONTENTS.

ARTICLES:—	PAGE
Kopfer's Method for the Determination of Carbon and Hydrogen, by James J. Dobbie M.A., D.Sc., and Alexander Lauder	215
Crystallised Carbon Dioxide, by A. Liversidge, M.A., F.R.S.	216
Liquefaction of Hydrogen and Helium	216
Separations from Chromic Acid, by Harry Brearley	216
The Special Analysis of Non-conducting Compounds by means of their Melted Salts, by A. de Gramont	218
On the Spectrum and the Nature of Neodymium, by Eug. Demarcay	219
On the Yttric Earths contained in the Monazite Sands, by P. Schützenberger and O. Boudouard	220
A Revision of the Atomic Weight of Zirconium, by F. P. Venable	221
PROCEEDINGS OF SOCIETIES:—	
THE ROYAL SOCIETY	223
ROYAL INSTITUTION	223
NOTICES OF BOOKS.—The Workmen's Compensation Act, 1897—General Elementary Science—The Gas Engineer's Pocket Book—First Year's Course of Experimental Work in Chemistry—An Elementary Course of Practical Organic Chemistry	223
CHEMICAL NOTICES FROM FOREIGN SOURCES	224
MEETINGS FOR THE WEEK	226

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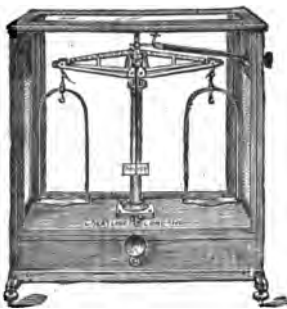
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THE CHEMICAL NEWS.

Vol. LXXVII., No. 2007.

KOPFER'S METHOD FOR THE DETERMINATION OF CARBON AND HYDROGEN.

By JAMES J. DOBBIE, M.A., D.Sc., and ALEXANDER LAUDER.

HAVING had occasion recently to try some of the methods of determining carbon and hydrogen which have been proposed from time to time as alternatives to those in common use, we believe that the results which we have obtained may be of sufficient importance to those who are interested in organic analysis to justify their publication.

Of the methods which have been proposed to replace the ordinary method of combustion with copper oxide, that devised by Kopfer (*Zeitschrift für Anal. Chemie*, 1878, xvii., 1) is perhaps best known. It is described at considerable length in such recent works as Vortmann's "Anleitung zur Chemischen Analyse Organischer Stoffe," and in Meyer and Jacobsen's "Lehrbuch der Organischen Chemie" (1893) it is referred to as a method which has been favourably criticised in many quarters.

Great advantages are claimed for this method by its author, of which the most important are that the combustion-tube may be much shorter than that usually employed; that the amount of gas consumed is less, and

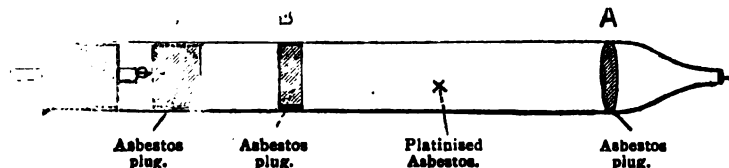
tube, before being used, is moderately ignited in a current of oxygen for half an hour. It is then allowed to cool, the absorption apparatus attached, and the boat containing the substance introduced into the part of the tube lying between the plugs a and c. A slow stream of oxygen is now passed through the tube, which is then heated, beginning with the three front jets under the layer of platinised asbestos, the flames being so regulated that, at the most, they are 4 c.m. in height. After some minutes the back movable burner is also lighted and placed under the plug c. The flame may be large, and may reach 2 c.m. over the tube. The movable flame is gradually brought nearer to the substance, whereby the progress of the combustion, which can be observed by the gas-bubbles passing through the potash solution, can be regulated. Lastly, the fourth gas burner under the front part of the tube is also lighted, and the operation brought to an end in the same way as in an ordinary combustion.

The platinised asbestos used is prepared by shaking up platinum-black with woolly asbestos.

The process requires to be modified when other elements besides carbon, hydrogen, and oxygen are present.

Kopfer gives numerous results obtained by his method. With cane-sugar he found, in two experiments, results agreeing perfectly with the calculated quantities, each combustion taking only one hour. With strychnine, a body containing a high percentage of carbon, three analyses gave results agreeing with one another and with the theoretical numbers. Each combustion in this case occupied one and a half hours.

For substances which are volatile without decomposition an additional layer of platinised asbestos, 50 c.m. long, has to be employed. With this additional layer, and some slight modification of the manipulation, Kopfer succeeded in obtaining excellent results with such sub-



the time occupied in the operation shorter. It is further claimed for the method that it gives more accurate results for hydrogen, and that it is applicable to all classes of organic bodies. The results given by the author for substances differing so widely from one another in character as naphthalene, para-dichlorobenzene, nitro-benzene, and allyl-mustard oil, leave, as a rule, nothing to be desired in point of accuracy.

In view of the manifest advantages supposed to belong to this method, it seemed to us somewhat surprising that it had not come more generally into use. With the view of testing its capabilities, we made a series of determinations, following as closely as possible the directions given in the original paper.

The combustion is carried out in a hard glass tube 40 to 50 c.m. long and from 13 to 15 m.m. wide, which is drawn out at one end to about 4 m.m. diameter.

A plug of asbestos, A, covered with thin platinum foil, is placed in the drawn out end of the tube, and behind this from 20 to 25 c.m. of the tube are fitted with platinised asbestos held in position by a second plug, B, which ought not, however, to fit the tube too tightly. A third plug, C, is necessary, which ought to be cylindrical and to fit the tube properly, and be provided with a loop of platinum wire, so that it may be conveniently drawn out and in. The whole tube is placed in a furnace of special construction provided with four fixed burners in the front part and with a movable burner behind, and that part of the tube which contains the platinised asbestos is wrapped in three or four plies of iron or copper wire gauze, in order to protect it from the gas flames. The

stances as naphthalene, toluene, and xylene; each combustion occupied from 1½ to 1¾ hours.

In dealing with bodies containing nitrogen a longer tube—about 60 c.m.—is employed, and a layer of granular lead peroxide is placed in the front part of the tube, which is kept at a temperature of 180 to 200° C. during the combustion. The analysis of nitro-benzene performed in this way also gave satisfactory results.

In the analyses which we carried out by this method every care was taken to follow closely Kopfer's directions, and to employ the various precautions which he indicates as necessary in the case of certain classes of substances. In order to secure the most favourable conditions for complete combustion, we prepared the platinum-black by Loew's method (*Berichte*, xxiii., 289a), which is considered to yield this substance in a very active condition.

Our first experiments were made with easily combustible substances, and after a little practice we obtained fairly accurate results. The following numbers were obtained with cane-sugar:—

- I. 0.2058 grm. gave—
0.1168 grm. H₂O = 6.30 per cent hydrogen.
0.3153 grm. CO₂ = 41.78 " carbon.
- II. 0.2190 grm. gave—
0.1220 grm. H₂O = 6.18 per cent hydrogen.
0.3335 grm. CO₂ = 41.53 " carbon.

	Found.	Calculated.
Carbon ..	41.78 p.c.	41.53 p.c.
Hydrogen..	6.30 " "	6.18 " "
		6.44 "

Time occupied in both cases about one hour.

With volatile bodies we were less fortunate; with ethyl alcohol and benzene we failed altogether to obtain results even approximately correct.

As a nitrogenous body not difficult to burn we selected urea; the combustion was carried out with the modifications indicated above. We obtained the following results:—

0.2085 grm. gave—	
0.1210 grm. H_2O = 6.46 per cent hydrogen.	
0.1492 grm. CO_2 = 19.51 " carbon.	
	Found. Calculated.
Carbon .. = 19.51 p.c.	20.00 p.c.
Hydrogen .. = 6.44 "	6.66 "

With substances containing a large percentage of carbon the results were altogether discouraging. Cinchonine and corybulbine (one of the alkaloids of *Corydalis cava*) were analysed, but the results were invariably low.

After spending much time and trouble over the method, and after performing a large number of experiments, of which we have not thought it worth while to quote more than a few, we came to the conclusion that the method is useless as a general method for the determination of carbon and hydrogen. With substances which are non-volatile and easily burned, it can be made to yield fairly accurate results. In the case of volatile substances we believe it to be altogether inapplicable, the difficulty of so regulating the rate of combustion as to prevent any of the material from volatilising undecomposed into the absorption apparatus being insuperable. It is well known that by the usual methods it is occasionally extremely difficult to effect complete combustion of some of the alkaloids. We had hoped to find this method applicable to such cases on account of the energetic oxidising action attributed to the platinum-black. As a matter of fact, we found it inferior to the ordinary method, the results being invariably too low. Others who have attempted to apply the method to such substances have also found it wanting. Zeisel, in a paper on Colchicin (*Monatshefte*, vii., 1886, p. 557), obtained results which were too low both for carbon and hydrogen.

We do not mean to assert that we have demonstrated that the method is absolutely incapable of yielding accurate results except with easily combustible bodies like cane-sugar and urea. If, however, it is capable of a more general application, our experience shows that the conditions for success would have to be specially studied in each case. A good practical method for determining carbon and hydrogen should be applicable with very slight modification to all the cases that may arise, and this condition is admirably fulfilled by the copper oxide method as usually practised. Since Kopper's method does not fulfil this condition, it is manifestly of little practical value to the organic chemist.

We have also investigated some of the other methods which have been proposed for the estimation of carbon and hydrogen, especially Lippmann and Fleissner's method with copper oxide asbestos, and hope to communicate the results in a future paper.

University College of North Wales, Bangor.
April 15, 1898.

CRYSTALLISED CARBON DIOXIDE.

By A. LIVERSIDGE, M.A., F.R.S.,
Professor of Chemistry, University of Sydney, New South Wales.

WHEN solid carbon-dioxide is examined under the microscope it presents along its edges projecting wire-like crystals, which have branching filaments issuing from them, apparently at right angles, resembling somewhat the groups of minute crystals seen in crystallised iron, gold, and ammonium chloride.

The rapidity with which the carbon-dioxide evaporates makes it difficult to catch the form of the crystals, either by photography or other means.—*Proc. Australasian Assoc. for the Advancement of Science.*

LIQUEFACTION OF HYDROGEN AND HELIUM.

ON Thursday, the 12th inst., Professor Dewar gave to the Royal Society an account of the results of recent low temperature research, which have culminated during the last day or so in the successful liquefaction of hydrogen and helium. About 20 cubic centimetres of clear colourless liquid hydrogen were obtained, and its properties and reactions examined.

SEPARATIONS FROM CHROMIC ACID.

IV. THE SEPARATION OF CHROMIUM,

AND

A REPLY TO MR. GALBRAITH'S CRITICISMS.

By HARRY BREARLEY.

THE reaction between potassium chromate and a neutral chromic salt has been so frequently observed, and analyses of the resulting precipitate so often recorded, that the fact of its existence needs only to be casually mentioned. The reaction, expressed in a manner analogous to the preceding ones, is very fully dealt with in Storer and Eliot's papers (*CHEM. NEWS*, vi., 121).

The bearing of this reaction on the quantitative separation of the salts of chromic oxide from chromic acid is just as important here as in the like separation of iron or aluminium. In practice, however, the former separation is so rarely necessary that one seeks almost in vain for an account of processes in which to note possible interferences.

In making the final analyses of the precipitated $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$, Storer and Eliot made use of a method by Vogel (Fresenius, "Quant. Anal.," p. 294), but the preliminary analyses were effected by dissolving the precipitate in nitric acid and separating the chromic oxide with a faint excess of ammonia. Separations by these means of the usual amount of potassium chromate from half a grm. of chromium (as Cr_2Cl_6) showed that (using fractional filtration) as little as 50 per cent of the chromic acid might be in the filtrate, the deficiency being presumably due to the formation of $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$, or a more basic chromate. Storer and Eliot remark that the precipitate obtained in this way is brownish green in colour, but after thorough washing assumes the common appearance of chromic oxide. Without commenting on the worthlessness of the colour as an indication of purity, or on the fact that Storer and Eliot repeatedly note the impracticability of completely decomposing $\text{Cr}_2\text{O}_3 \cdot \text{CrO}_3$ by washing, is it not strange that when this method of analysing the precipitate comes to be abandoned there should be urged against it such comparatively insignificant errors as the solubility of the chromic hydrate in the excess of ammonia, whilst the error due to the formation of $x\text{Cr}_2\text{O}_3 \cdot y\text{CrO}_3$ should be entirely overlooked?—another striking instance of the assumption that a compound so feeble as to be decomposed by washing must needs be completely resolved by an excess of an energetic precipitant.

Failing to meet with already described methods of precipitating the chromic chloride from the potassium chromate, it seemed interesting to observe with what success some common methods of precipitating chromic salts from pure solution could be applied to this separation. As these observations were very much in line with those already illustrated in preceding separations, it will suffice to state the conclusions in general terms.

Separation with Soda Hydrate.

With this reagent in slight excess, and the mixture merely raised to boiling, there was an approximate perfect separation. The prolonged boiling, which is necessary

to re-precipitate small amounts of chromic hydrate, is decidedly an advantageous operation. It transforms the separation into a perfectly accurate one. Larger amounts of soda precipitate the chromic hydrate free from chromic acid without prolonged boiling, but they are inadmissible on account of the solubility of the precipitate in an excess of the precipitant.

Separation with Soda Phosphate.

This reagent may be applied in two or three ways. First alone, in which case—presuming that there is free acid to be neutralised—it becomes interesting to note that the salts of chromium, like those of iron and aluminium, but in a less degree, are able to dissolve their own hydrate, and roughly indicate the point at which they begin to do so by changing to an olive-green colour. If to a solution thus neutralised a small excess only of soda phosphate be added, the results are 1 or 2 per cent too low, a deficiency which may be amended by adding larger excesses. From a faintly acid solution the chromic acid is perfectly separated by adding soda phosphate and soda acetate, and boiling ("Select Methods," p. 127). It is noticeable in these cases that when the precipitate forms in the cold it is frequently dark-coloured, but changes to the accustomed green as the temperature rises. This would seem to supply a reason for maintaining the solution at boiling-point for a while, and to indicate that the reagents had best be added to the hot solution. The phosphate may be added to an acid solution, and then caused to precipitate the chromium by making the solution alkaline. A slight excess of ammonia along with minimum amounts of phosphate will not do,—one or the other reagent must be decidedly in excess. Soda along with soda phosphate gives excellent results. Other methods in which the chromic acid is precipitated have not been observed.

Oxidising Reagents.

Chromium salts are frequently used to illustrate the powers of a new oxidising agent, and so it happens that a longer list of substances than may be appropriately named are said to be capable of oxidising chromic oxide to chromic acid; yet, strangely enough, a great number of these reagents are elsewhere said to partly fail when applied to such a purpose. The anomaly seems to be explained by the fact that the conditions under which the best results can be obtained are empirical ones, and may be easily missed in a casual test. Such, at least, has been my experience. Without attempting to state the order in which the oxidising reagents are to be preferred for this purpose, it will serve to illustrate the empirical nature of the general reaction if I carefully examine one reagent. I have chosen—

Permanganate in Acid Solutions,

under which head may be included the freshly precipitated manganese dioxide.

The oxidising effect of permanganate is not immediate, but gradual, and the last portions of the chromic oxide strongly resist the attacks of small excess of permanganate, or rather of the dioxide got from the decomposed permanganate. Thus, working on 25 c.c. of reduced chromate, we may recover after boiling for—

5	10	30	60 minutes,
21'1	21'9	23'3	23'9 c.c.

of the added chromate, and it would seem to be possible to complete the oxidation by boiling long enough. I have oxidised 49.7 c.c. out of 50 c.c. of reduced K_2CrO_4 , when the permanganate, being added from a burette, was never more than a few drops in excess (permanganate = 25 grs. per litre).

The acidity (H_2SO_4) of the solutions is another factor. Increasing acidity favours the oxidation, but this modification cannot be worked very far, because, according to Leffler (CHEM. NEWS., lxxvii., 156), the precipitated manganese

oxides become partly dissolved without being completely decomposed, and are thus able to augment the measured oxidising effect of the chromic acid, and further (Arnold, CHEM. NEWS., lxxi., 292) the acid may become so strong that "the complete oxidation of Cr_2O_3 to CrO_3 is practically impossible."

The permanganate may be added in too great an excess. This is an item of general knowledge. Experiments have been described in which 20 per cent of the whole bichromate goes down with the manganese oxides on this account. On the other hand, one may use too little, and fail to complete the oxidation. The addition of permanganate in such excess as to retain a rose tint after boiling a certain number of minutes cannot be accepted as a universal rule, because the production of the rose tint may be so far delayed, by the presence of other elements, that too great a bulk of manganese oxides is accumulated before it can be said to persist at all.

I must explain here that this work on the oxidising effects of permanganate has been done since stating my objections to Mr. Galbraith's process for estimating chromium in ferrochromium, and being thereby led to a justification of our respective positions, I propose to notice Mr. Galbraith's letter in this connection.

The letter (CHEMICAL NEWS, vol. lxxvii., p. 187) is essentially made up of the suggestion that I have misrepresented his paper of 1893 and some very satisfactory test analyses in support of the soda modification.

It has been objected to Galbraith's original process that the sample was not altogether soluble in dilute sulphuric acid. This, along with the low results due to using large excesses of permanganate, are the objections to which I refer as having been examined into in Mr. Galbraith's paper (1893).

The only reason I can offer for having failed to announce prior to 1893 that the large excess of permanganate could be reduced with advantage is that it was a point acted upon in the laboratory where I was trained and was treated as a matter of general knowledge. Moreover, I did not discover it, and even if I had, surely Mr. Galbraith, who kept his knowledge of the improvement hid away for fifteen years (1878–93), ought not to chide me for failing to publish a fact which those around me—older and more experienced—regarded as no novelty.

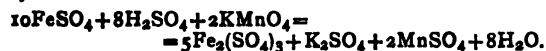
I cannot imagine why, in the fourth paragraph of Mr. Galbraith's letter, he makes a point of his having "tried the effect of the presence of iron" in connection with some other method. Mr. Galbraith's clear statement of my objection in paragraph 7 shows him to have understood it to refer only to the soda modification. As I made no criticism whatever of the 1873 process, my objection can in no way be supposed to refer to it.

It is pleasanter to notice that in the latter part of his letter Mr. Galbraith has given the satisfactory tests which he omits from his original (1893) paper, and which, I am happy to say, I can now broadly confirm, as also I can justify my bad results.

I do not presume to blame Mr. Galbraith, and at the same time I claim to be acquitted for having failed to see—or, at least, to state—clearly the precise rôle which the manganous salts play in the operation. The instructions are to dissolve the sample in sulphuric acid (1:6) and add permanganate. Mr. Galbraith may add as much permanganate as ever he can get in without causing a permanent rose tint; I add much less, and yet sufficient to completely oxidise the chromic oxide; another operator may add even less than I do. So far, all are right; Mr. Galbraith gives no instructions on this point, although he is admittedly aware (CHEMICAL NEWS, vol. lxxi., p. 316) that the excess mentioned in 1877 has been generally thought too much, and variously reduced. When these varying amounts of permanganate—all sufficient to completely oxidise the Cr_2O_3 to CrO_3 —lead, after treatment with soda, to varying recoveries, am I not right in my first contention, viz., that the process needs stating in more definite terms before it can be satisfactorily worked?

Mr Galbraith's letter seems to imply that he believes his accurate results to have been obtained from a soda treated solution containing manganous salts. I can now show that this is not so, except in a small degree, and as there exists no statement on the matter to which I can refer, I beg to submit the following explanations:—

To begin with, the equation on p. 132 should be replaced by—



Considering now the behaviour of the manganous sulphate only, we know that when permanganate is added to a neutral solution of a manganous salt they are mutually decomposed. This is the principle of the Volhard and Guyard methods of estimating manganese, and the emphasis laid on the neutral or nearly neutral state of the manganous solution is perhaps accountable for one's overlooking the possibility of such a reaction taking place in the strongly acid solution used in dealing with chrome irons. Such a reaction, however, is possible, and is explanatory of the larger precipitate which in such cases precedes the rose tint.*

Is it possible now to completely precipitate the manganous salt formed in the ferro-chrome or steel solution by adding permanganate? If it can be done—and Mr. Galbraith, however unconsciously, has habitually pushed his oxidation to that point—then we have the explanation of the success of the method in his hands.

Let the matter be tested experimentally by adding permanganate to the formation of a faint precipitate of the manganese oxide, and then adding varying amounts of permanganate in excess. If the increasing amounts of permanganate more and more completely precipitate the manganous sulphate, the manganous hydrate to be precipitated becomes less and less, and hence the reducing action complained of less evident. Two grms. of iron were taken in each case, along with 50 c.c. K_2CrO_4 (1 c.c. = 0.01 gm. Fe). The solutions were boiled for fifteen minutes after adding the final permanganate, and fractional filtration was employed. Strength of permanganate, 25 grms. per litre. A decided excess of soda was used in each case.

Permanganate to faint precipitate. C.c.	Excess of permanganate. C.c.	C.c. K_2CrO_4 recovered.	Percentage recovery.
28	7	23.12	46.2
28	17	38.6	77.2
28	27	48.2	96.4
28	29	49.5	99.0
28	29	49.9	99.8
28	31*	49.95	99.9

In the last test, marked with an asterisk, the permanganate was not completely decomposed; the result is obtained by treating the filtered fraction with HCl according to Stead's method.

This table supports Mr. Galbraith's results. At the same time it confirms everything I have said, and, pointing out as it does so serious a source of possible error, will, I hope, be received as a friendly contribution. I have only to remark that the continued boiling with the amounts of acid used above (30 c.c. dilute, 1 to 4, H_2SO_4 for 2 grms. Fe) did not worsen the results as would have been evident if the manganese oxides were soluble (as Mr. Leffler finds them to be) in larger amounts of acid.

Mr. Galbraith raises an interesting point in connection with those assays which are allowed to stand for a long time. It does not appear how the manganous salts could be responsible for the error in cases where they had been completely oxidised by the permanganate. May I ask for some further assurance that the error is not due to the

causes just pointed out? The manganous sulphate and permanganate reaction in an acid solution is completed only after considerable boiling, and it is quite possible to obtain the rose tint, and then fall short of the complete oxidation of the manganous salt.

Returning to the general behaviour of permanganate, I may say that Stead's modification (*Journ. Iron and Steel Inst.*, 1893, vol. i.) of the acid permanganate process is less subject to incidental errors than any other modification of the process I know. It consists in decomposing the permanganate and manganous oxide by dilute HCl. This not only avoids the error, according to design, of a large precipitate, but by the oxidising effect of the liberated chlorine it also minimises the misfortune of having added too small an excess of permanganate.*

We say that the large excess of permanganate carries down some of the chromic acid—which, to be sure, like many another such an expression, is not a very satisfactory way of accounting for the deficiency, however well it may prepare us to expect it. This carried-down chromate would seem to be more than in contact with the manganous oxides, since we can neither remove it by washing (in reason), nor account for it by filtering fractionally. We are told that the washings from these large precipitates are never absolutely colourless, and that the bond, whatever it is, can be loosened by treating the mixture with soda hydrate. Such characteristics have all along presented themselves in connection with the basic chromates whose interference with separatory methods we have been considering. Does not the coincidence suggest that we may have here another such interference?

Totley, near Sheffield.

THE SPECTRAL ANALYSIS OF NON-CONDUCTING COMPOUNDS BY MEANS OF THEIR MELTED SALTS.

By A. DE GRAMONT.

THE natural silicates, reduced to a fine powder and dissolved in a pearl of a melted alkaline carbonate, through which the disruptive discharge of a condenser charged by an induction coil strikes, have given me in the spectro-scope all the principal lines of silicon (*Comptes Rendus*, Jan. 25, 1897). The greater number of the elements composing or accompanying the silicates also show their presence in a similar manner. I have thus been led to search, in this operation, for a method, generally applicable to non-conducting minerals, which cannot be directly analysed by means of their spectra in the manner already described (*Comptes Rendus*, July 8, 1895).

The continuous and very bright appearance of the line spectra of the components of the minerals studied bring these latter under the conditions of the examination of melted salts, of which I have but recently published the dissociation spectra. Not only the silicates, but the sulphates, sulphides, fluorides, carbonates, oxides, and—apart from minerals—the insoluble non-conducting precipitates, obtained successively in analytical operations by the wet way, all give satisfactory results by this method of investigation. As in the case of all spectral reactions, the sensitiveness differs notably between one simple body and another; it is, however, better as a rule than that of the blowpipe or the wet method.

This method only requires a very small quantity of material, which must be finely ground in an agate mortar, and intimately mixed with the salt destined to disintegrate

* A little manganous sulphate added to a solution overdosed with permanganate readily decolorises it. This seems to be a better way than using more H_2SO_4 , as recommended by Galbraith; it may sometimes usefully replace alcohol in that office.

* It should be noted that, owing to the presence of large amounts of manganous salts, the ferricyanide test may be falsified by the production of a reddish brown precipitate. This precipitate, as may be seen by viewing a blue solution against a reddish paper, not merely by clouding the test drop, but by neutralising the colour also (*Chem. News*, Lxxvii, 133).

and dissolve it. A small quantity of this mixture is melted in a flame, on the flattened end of a stout platinum wire forming the lower side of the <-shaped arrangement I have previously described, where the apex of the angle is the point where the spark strikes when the two poles are brought close to each other; these poles are connected to the poles of the Leyden jar and the coil.

The melted salt used as the igneous solvent of the mineral has generally been carbonate of lithium, on account of the simplicity of its spectrum, its easy fusibility, and the rapidity of its solvent action on the silicates. Carbonate of sodium gives good results, but it is less fusible, and the maximum of intensity of the spectrum obtained by its use is considerably slower in showing itself than with Li_2CO_3 . The two salts have been used concurrently in most of the experiments, and the superiority of the dissolving power of the lithium salt, at a relatively low temperature, appeared to me to warrant its use in all cases of the analysis of a silicate by igneous disintegration.

The small number of the carbon lines, of which I have already described both the position and the appearance in the carbonates (*Comptes Rendus*, July 29, 1897), is in practice reduced to two—657.8 in the red and 426.7 in the indigo. The spectrum of the solvent thus shows very few lines, and, instead of interfering with the observations, they serve as additional data for locating the position of those sought for.

This method offers another advantage, viz., the absence of the air spectrum, which only appears after the volatilisation of the melted salt, or when the flame does not properly envelope it.

The use of other salts has been tried. Boric acid and the borates appeared to be very bad conductors, whether melted or in the state of vapour; the spark passes with difficulty, giving both the spectrum of platinum and of air. Further, the lines of boron can only be obtained under special conditions, above all with regard to the strength of the spark used. There is, therefore, no advantage to be gained by using salts of boron as a solvent, nor is it easy to detect boron by its spectrum of dissociation; it is better, on the contrary, to depend on the well-known green bands it gives in the flame.

We can, on the other hand, make use of the alkaline bisulphates, either for detecting the metals (but we must not go beyond a slight condensation, insufficient to cause the appearance of the sulphur lines), or for studying the most refrangible part of the spectrum, where no sulphur lines are to be found.

The source of heat to be used varies with the salt; for Li_2CO_3 an ordinary spirit lamp will suffice, but with the other carbonates or with the bisulphates it is necessary to use a Bunsen burner or a petroleum oil-lamp.

The general arrangement of the apparatus, and the method of making the observations, is the same as that which I adopted when studying the spectra of melted salts. The condensation only is varied; it has been reduced to one jar, in the case of the metals only, without causing the lines of the metalloids to appear. If we have any fear of the formation of reducible metallic compounds which might attack platinum, the lower platinum spatula may be replaced by a morsel of pure Siberian graphite, hollowed out like a small crucible, to contain the drop of melted mixture, and held in the flame by a pair of platinum-tipped forceps.—*Comptes Rendus*, vol. cxvii., No. 16, 1898.

praseodymium vary considerably, and that these variations prove the existence of several distinct simple bodies (according to some authorities, there are as many elements as there are absorption-bands). But the proof of these variations has never yet been shown, nor has any work been published with the object of establishing the truth or the falsity of the statement. I decided to submit a considerable quantity of didymium, freed from lanthanum and cerium, to a series of fractionations by the method of ammoniacal nitrates (Auer). They gave me a double nitrate which, when melted in its own water of crystallisation and observed through a thickness of more than 0.1 metre, no longer showed any trace of the principal band of praseodymium. This nitrate was the starting-point for a fresh lot of fractionations which enabled me to eliminate, on the one hand, samarium and the neighbouring earths in notable quantities, and, on the other hand, a slight trace of praseodymium. I thus obtained more than twenty successive portions which exhibited the most striking identity from every point of view. The praseodymium was invisible through thicknesses of more than 0.1 metre of the melted nitrate; the samarium could not be detected either by its absorption bands near H, usually so intense, or by its strong ultra-violet rays in the spark spectrum, or in its fluorescent spectrum in sulphate of calcium.

To convince myself that, in spite of the identity of the spark spectra of the first and last portions, there was no earth present not having an absorption spectrum or a discernible spark spectrum, I had recourse to a spectrocolorimeter. The result was negative; both portions showed the same absorption-spectra with the same intensity; it was impossible to detect the slightest difference between any of the bands. In my opinion, therefore, we must conclude that, contrary to the statements of several savants, neodymium is a simple body, and not a mixture of elements. Several very much shorter series of fractionations enabled me, in fact, to observe the spectra of samarium and of praseodymium individually rapidly increasing and diminishing. I may add that neodymium from entirely different sources (cerite, samarskite, mosandrite) has always given me, when properly purified, identically the same spectrum. The opinion that neodymium is composed of earths incomparably more difficult to separate than no matter which other group of elements among the rare earths, is thus shown to be devoid of experimental foundation. Other methods of fractionation have led me to the same results, though in a much slower manner.

Oxide of neodymium, prepared from the oxalate by prolonged calcination in the air, is not grey, greenish, or rose coloured, as various authors have stated, but of a clear bright blue colour, with lilac reflections in the mass. Very small traces of praseodymium, samarium, or terbium affect the colour to a marked degree; this is therefore a very good indication of its purity. The colour of the salts of neodymium is of a lilac-blue through only a slight thickness; through a medium thickness, it is a rose-violet; in concentrated solutions, through a thick layer, it is of a very rich reddish violet colour. Even small proportions of samarium or of praseodymium suffice to change the colour to a more or less yellow or brownish red, such as has always been described as the colour of neodymium up to the present time.

In the form of chloride (a 10 per cent solution of the oxide in HCl, through a thickness of 0.036 metre) neodymium gives the following bands:—

732.4	
691.0	
680.4	Strong.
673.1	Faint.
637.3	
629.2	Very faint double.
623.4	
578.3	About the middle of the very strong band.
532	About the middle of the strong band.

ON THE SPECTRUM AND THE NATURE OF NEODYMIUM.

By EUG. DEMARCAV.

SINCE the important work done by M. Auer von Welsbach (*Monats. f. Chem.*, 1885, p. 477), it has been announced that the number of lines in the spectra of neodymium and

522	Fairly strong.
510.9	Very strong in the middle.
476.8	Strong.
469.1	"
462.4	Cloudy, diffuse, fairly strong.
435.1	Faint.
429.4	Very faint.
428.1	Strong.
420 (about)	Very faint.

The whole of the first portion of this spectrum, up to 510.9 inclusive, is identical with that described by M. Auer von Welsbach (*loc. cit.*). But this *savant*, out of all the following lines, only mentions 428.1. I cannot easily understand the omission of the lines 476.8 and 469.1 (very near to a praseodymium band, but certainly not due to it), and 462.4, which are all strong, and can be easily seen even in a fairly weak solution. The presence of a certain amount of samarium, as shown by M. Lecoq de Boisbaudran, causes the blue bands I have just mentioned to disappear in a vague and hardly visible nebulosity. Is this perhaps the reason of their omission by M. Auer and, more recently, by M. Brauner (*Chem. Soc. Trans.*, 1898)?

The spectrum varies enormously in certain bands with the nature of the acid present. For example, the small group towards 630 is unrecognisable in nitric solution. M. de Boisbaudran has already noticed several analogous facts.

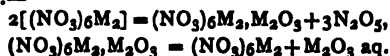
I will finish by expressing the hope that chemists making observations of absorption-spectra will, in future, describe accurately the exact composition of the solution and the thickness of the layer through which it has been examined: without these data, we cannot well interpret the results published in many papers.—*Comptes Rendus*, cxxvi., No. 14.

ON THE YTTRIC EARTHS CONTAINED IN THE MONAZITE SANDS.*

By P. SCHÜTZENBERGER and O. BOUDOUARD.

THE pure yttric oxides, after being converted into nitrates, are submitted to partial decomposition at a temperature of from 315° to 325°. The operation is carried on in a cylindrical platinum crucible plunged by any convenient means into a bath of nitrates of potassium and sodium in equal parts. The melted nitrate commences by giving off nitrous vapours, it then gets pasty, and finishes by forming a crystalline mass, solidifying at 315°. When all decomposition appears to have finished, it is allowed to cool, and is then treated with boiling water. The mass divides into an insoluble sub-nitrate and a soluble part consisting of a neutral nitrate; this latter, evaporated to dryness, is submitted to the same treatment, and gives a fresh insoluble sub-nitrate and a soluble neutral nitrate. The sub-nitrates thus obtained are washed with warm water and transformed into sulphates, and the atomic weight of each one is determined by a prolonged calcination of these sulphates at a bright red heat.

It is a very easy matter to establish a theory for this decomposition of the yttric nitrates at the temperature of 325°; it should be effected according to the following reactions:—



Adding these two equations member to member we get—



Each partial decomposition would thus separate the half of the oxide entering into the reaction. In the numerous experiments which were made, the weight of the oxides

obtained often represented one-third of the oxide used, and several times a half. This variation certainly points to this, that the formation of crystalline crusts during the decomposition prevents this latter from being complete. But if we allow the decomposition to proceed for a sufficient length of time, we always obtain the half of the oxide which has entered into the reaction.

Results Obtained.—We shall now give some of the series of fractionations which have appeared to us to be of the greatest interest. The two methods—that of fractional crystallisation and that of fractional fusions—have, further, been combined.

A.—Two sub-nitrates were made, and each separate portion has been submitted to a fractional crystallisation.

Sub-nitrate No. 1.

This sub-nitrate, after having been transformed into nitrate by simple solution in nitric acid, was itself partially decomposed into two other subnitrates.

SUB-NITRATE NO. 1 (1).

First Crystallisation.

Atomic weight		123.2
Anhydrous sulphate used	..	3.6140
Oxide obtained		1.9910

Second Crystallisation.

Atomic weight		122.25
Anhydrous sulphate used	..	2.8145
Oxide obtained		1.5460

SUB-NITRATE NO. 1 (2).

First Crystallisation.

Atomic weight		114.7
Anhydrous sulphate used	..	3.7790
Oxide obtained		2.0260

Second Crystallisation.

Atomic weight		100.85
Anhydrous sulphate used	..	4.7238
Oxide obtained		2.4138

Sub-nitrate No. 2.

First Crystallisation.

Atomic weight		102.4
Anhydrous sulphate used	..	2.7330
Oxide obtained		1.4020

Second Crystallisation.

Atomic weight		100.1
Anhydrous sulphate used	..	2.5250
Oxide obtained		1.2840

Mother-liquors from Sub-nitrate No. 2.

First Crystallisation.

Atomic weight		100.9
Anhydrous sulphate used	..	4.7230
Oxide obtained		2.4095

Second Crystallisation.

Atomic weight		96.0
Anhydrous sulphate used	..	2.5625
Oxide obtained		1.2815

Third Crystallisation.

Atomic weight		94.0
Anhydrous sulphate used	..	2.3482
Oxide obtained		1.1645

The sulphate (at. wt. = 96) was submitted to a fractional crystallisation:—

First Crystallisation.

Atomic weight		98.1
Anhydrous sulphate used	..	3.4410
Oxide obtained		1.7350

Second Crystallisation.

Atomic weight		93.0
Anhydrous sulphate used	..	2.1925
Oxide obtained		1.0825

* *Bull. Soc. Chim.*, Series 3, vol. xix.-xx., No. 6.

The sulphate (at. wt. = 98.1), after having been calcined, was again transformed into nitrate and sulphate. Two more crystallisations were made:—

First Crystallisation.

Atomic weight	99.65
Anhydrous sulphate used ..	3.2980
Oxide obtained	1.6740

Second Crystallisation.

Atomic weight	94.5
Anhydrous sulphate used ..	2.1037
Oxide obtained	1.0473

The first crystallisations of these two fractionations (at. wts. = 98.1 and 99.65) were united and submitted to a fresh fractional crystallisation:—

First Crystallisation.

Atomic weight	101.1
Anhydrous sulphate used ..	5.5187
Oxide obtained	2.8172

Second Crystallisation.

Atomic weight	96.7
Anhydrous sulphate used ..	3.7020
Oxide obtained	1.8565

The sulphate (at. wt. = 101.1) was again separated by two fractionations:—

First Crystallisation.

Atomic weight	102.35
Anhydrous sulphate used ..	4.5607
Oxide obtained	2.4320

Second Crystallisation.

Atomic weight	98.0
Anhydrous sulphate used ..	4.3135
Oxide obtained	2.1750

The oxide which gave on analysis 102.35 was added to the oxide resulting from the calcination of the corresponding sulphate, and was transformed into nitrate and sulphate by making use of quite a fresh method of procedure. Before evaporating to dryness, the acid mother-liquor which remained over the crystals was decanted; the crystals were then gently calcined and, after dehydration, dissolved in water. This was crystallised and separated into two portions:—

First Crystallisation.

Atomic weight	104.1
Anhydrous sulphate used ..	3.5995
Oxide obtained	1.8590

Second Crystallisation.

Atomic weight	99.5
Anhydrous sulphate used ..	2.7840
Oxide obtained	1.4125

Acid Mother-liquors.

Atomic weight	102.6
Anhydrous sulphate used ..	2.4702
Oxide obtained	1.2682

The sulphate (at. wt. = 104.1) was separated into two fresh portions, which gave:—

First Crystallisation.

Atomic weight	104.55
Anhydrous sulphate used ..	2.1993
Oxide obtained	1.1375

Second Crystallisation.

Atomic weight	97.5
Anhydrous sulphate used ..	1.4055
Oxide obtained	0.7070

As is shown by the first results we have obtained at different times atomic weights represented by figures very close to 98. We have re-united all the oxides responding to this condition, and have made a fresh fractional crystallisation:—

First Crystallisation.

Atomic weight	97.1
Anhydrous sulphate used ..	2.3237
Oxide obtained	1.1675

Second Crystallisation.

Atomic weight	97.45
Anhydrous sulphate used ..	2.2422
Oxide obtained	1.1280

Third Crystallisation.

Atomic weight	97.7
Anhydrous sulphate used ..	3.1568
Oxide obtained	1.5900

Fourth Crystallisation.

Atomic weight	94.5
Anhydrous sulphate used ..	1.3310
Oxide obtained	0.6615

(To be continued).

A REVISION OF THE ATOMIC WEIGHT OF ZIRCONIUM.*

By F. P. VENABLE.

I. Purification of the Material.

PREPARATORY to entering upon the revision of the atomic weight of zirconium, a study was first made of the best method of decomposing zircons and securing pure preparations (*J. Anal. Appl. Chem.*, v., 551—554). The pulverised zircons were fused in nickel crucibles with sodium hydroxide and sodium fluoride. The melt was washed, dissolved in hydrochloric acid, and filtered from some of the silica and from undecomposed zircon. This filtrate was evaporated to dryness to render the silica insoluble, again dissolved in diluted hydrochloric acid, and filtered, and this was repeated two or three times. The further purification consisted in precipitation by ammonium hydroxide, washing, and re-solution in hydrochloric acid, followed by repeated crystallisations from boiling concentrated hydrochloric acid. After it had been thus purified it was found that traces of silica were still present. To remove these the chloride was dried, ignited, and the powdered zirconia was again and again treated with hydrofluoric acid. After driving this off, the zirconia was again melted with potassium hydroxide (purified by alcohol), taken up with hydrochloric acid, and subjected once more to crystallisation from strong hydrochloric acid. These crystallisations varied in number from twenty to thirty odd, and, if those which preceded the second fusion are to be taken into account, exceeded sixty in all. Just before using in the series of determinations of the atomic weight, this purified chloride was filtered by means of an unglazed porcelain suction-filter which had been cleaned by boiling for several days in strong hydrochloric acid, and then standing three days in fresh cold hydrochloric acid. The last acid was entirely free from colour. This filter was kept under distilled water when not in use. The filtration had to be carried out with hot solutions, but the liquid was not in contact with the filter more than five or six minutes.

When this work was undertaken it was believed that the substance under examination was zirconium tetrachloride. This belief was based on the work of Linnemann and upon my own determinations of the zirconia, these agreeing fairly well with the amount required for $ZrCl_4$. At the close of the determinations of zirconia recorded later, some determinations of the chlorine present were made, and were found not to agree at all with the amount requisite for the supposed formula. The matter did not seem easy of explanation, and the work was laid aside until time could be gotten for a more thorough study of

* From the *Journal of the American Chemical Society*, xx., No. 2.

this body. My study of the oxychlorides of zirconium has shown that there at least three:—

$\text{ZrOCl}_2 \cdot 3\text{H}_2\text{O}$ when crystallised from strong hydrochloric acid.

$\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ when crystallised from water.

$\text{ZrOCl}_2 \cdot 6\text{H}_2\text{O}$ when precipitated by hydrochloric acid from an aqueous solution.

All of these, dried in a stream of hydrogen chloride at 100° to 125° , have the formula $\text{ZrOCl}_2 \cdot 3\text{H}_2\text{O}$, and this water is lost only by heating from 180° to 210° .

The liquid filtered as above is clear and bright, and soon begins to deposit groups of needle-like crystals, or it crystallises to a solid mass if a large amount of the salt is present. In several cases the crystallisation was repeated two or three times from acid after this filtration, but no perceptible difference was made in the results. These crystals take up a considerable amount of acid which cannot be drained off. In this acid they easily melt, and if the excess is removed under a stream of hydrochloric acid gas the chloride can be gotten in the form of a dry white powder. The oxychloride can be thus dried without decomposition under a stream of hydrochloric acid gas at the temperature of 100° to 125° C. to $\text{ZrOCl}_2 \cdot 3\text{H}_2\text{O}$. As the difficulty in drying this salt was the main stumbling-block in the way of previous workers, interfering with its use for atomic-weight work, the removal of this obstacle seemed to place it among the most suitable of the compounds to be used for the purpose. A full account of the examination of the chlorides will be published shortly. Other compounds have been examined also in the progress of this work, and, though they may be used hereafter, they have seemed to me to present more difficulties than the chloride, and so this has been taken first.

Previous Determinations of the Atomic Weight.

It is perhaps best to give here a brief outline of previous determinations of the atomic weight of zirconium. Six such series have come under my notice. In three of these the sulphate was used, and in the others the chloride and the double fluoride of zirconium and potassium were used, and in one the selenate.

The determinations of Hermann (*J. Prakt. Chem.*, xxxi., 77), by means of the chloride, can be dismissed as untrustworthy, because of his failure to overcome the difficulties inherent in the use of the chloride as shown by Bailey's work and my own.

The work of Marignac upon the double fluoride I am not in a position to criticise properly, except in so far as to say that his analyses are not very numerous, and that they show a range of nearly three units in the atomic weight.

In 1825 Berzelius ignited the sulphate and gave six determinations of the ratio of the sulphate to the oxide. In some of the experiments he also precipitated the zirconium hydroxide by means of ammonium hydroxide, and determined the sulphuric acid in the filtrate by precipitation with barium chloride.

Mats Weibull also used the sulphate, and reports seven experiments with an entire consumption of 8.2335 grms. Bailey's own determinations number eight, using in all more than 16 grms. He gives full data as to his work, and it is well done and merits very careful attention. The following table is copied from his article (*CHEM. NEWS*, lx., 17). The figures have been re-calculated to the basis of O = 16.

	Mean.	Maximum.	Minimum.	
Zr : Cl	88.77	—	—	Hermann
ZrO_2 : HCl ..	90.14	90.98	89.29	Hermann
K_2ZrF_6 : K_2SO_4	90.53	92.80	90.06	Marignac
ZrO_2 : K_2SO_4 ..	90.64	91.26	90.24	Marignac
K_2ZrF_6 : ZrO_2 ..	90.8	91.3	89.9	Marignac
$\text{Zr}(\text{SO}_4)_2$: ZrO_2	89.45	92.65	89.27	Berzelius
$\text{Zr}(\text{SO}_4)_2$: ZrO_2	89.48	90.38	89.13	Mats Weibull
$\text{Zr}(\text{SO}_4)_2$: ZrO_2	90.65	90.78	90.46	Bailey

It is manifest that the determinations based upon the ignition of the sulphate are the only ones worthy of further attention. A brief criticism of these is necessary. First, as to Mats Weibull, Bailey says that the temperature used by him in freeing the sulphate from the excess of sulphuric acid was some 50° too low. This would of course give him variable and low results. Berzelius does not give exact data as to temperature used, but he seems to have heated the sulphate too high in driving off the excess of acid. Possibly more stress is to be laid upon the question of the purity of his sulphate and the correctness of the assumption that he had in hand the normal sulphate.

Bailey concludes from his experiments that the sulphate is stable up to 400° C., and that the excess of sulphuric acid can be completely driven off by the use of a temperature between this and 350° C. He further states that a mixture of the salt and free acid, as prepared by him, when heated to this temperature until constant, yields the normal sulphate. It must be said that he gives no proofs of this beyond the amount of zirconia found in his atomic weight determinations.

Whilst certain criticisms of the work of Bailey have occurred to me, I will refrain from mentioning them until I have had opportunity to repeat his experiments, and so make myself more familiar with the details of his method.

One criticism I can venture upon now, however. I doubt whether it is possible to ignite, without loss, zirconia along with ammonium carbonate, as was done by Berzelius and by Bailey to remove the "last two or three m.grms. of sulphuric acid." I have not ventured to use this method in getting rid of the chlorine which is held just as tenaciously as the sulphuric acid, as I feel sure that it could not be done without loss. Bailey adopted extraordinary precautions to prevent this loss, but it seems to me that it is not the currents of the external atmosphere, as he maintains, which are to be most avoided, but the mass of escaping vapour of the ammonium salts. It is easily possible for him to have lost several m.grms. of the finely-divided zirconia in his way, and, as he states, each m.grm. was equivalent to a variation of 0.25 in the atomic weight.

The Weighings.

In the following experiments the amounts of substance used varied from 1 to 5 grms. To avoid the disadvantage of a small error causing a large variation in the result, I would gladly have used larger amounts of the chloride, but many difficulties met me there. The purification of the zirconium chloride is slow and costly. It is best carried out in small portions of a few grms. at a time. Some 50 grms. have constituted the stock at my command. The drying of large portions and the subsequent ignition would be exceedingly tedious and time-consuming, besides requiring such apparatus as could not be well afforded. Five or six grms. have been about the largest amounts that could be well handled at one time. Even such an amount as that required from sixteen to twenty days for the completion of the experiment. It could not be safely hurried through in shorter time.

The weighings were carried out upon an excellently constructed Sartorius balance, intended for a load of 200 grms. The heaviest apparatus used weighed less than 60 grms. The weights were corrected by one which had been compared with the standard at Washington. All objects were weighed against a tare of as nearly the same size, form, and weight as possible, all of the flasks, crucibles, &c., being made in pairs. This partly avoided the necessity for a reduction of the weighings to a vacuum and corrections for moisture, pressure, &c. Such corrections would have had little meaning in comparison with the other inaccuracies of the process and manipulation, and could only serve to give a false appearance of excessive accuracy. The objects were left one half-hour in the balance-case before weighing, experiments having

shown that this time was sufficient. Of course the adjustment of the balance was carefully watched, and the balance, which has been used very little, was put to no other use during the progress of these experiments.

(To be continued).

PROCEEDINGS OF SOCIETIES.

THE ROYAL SOCIETY.

A CONVERSAZIONE was held at the Royal Society's Rooms at Burlington House on Wednesday last, the 11th of May, the guests and Fellows being received by Lord Lister and the Members of the Council.

Among the numerous exhibits we noted Professor Hele Shaw's Experiments on the Flow of Water, in which it is shown that stream-lines obtained in layers of inappreciable thickness have been found to be identical in practice with those arrived at mathematically.

Mr. Campbell Swinton showed some new forms of Crookes Tubes, the one of most interest being a fresh pattern of the experiment proving the physical movement of the molecules in a highly exhausted tube.

Dr. Armstrong had some attractive coloured photographs of those fascinating phenomena in the Yellowstone Park, U.S.A.

Close by were some excellent specimens of "Naturographs," produced by Dr. Sells's process of photography in natural colours. Three specially prepared negatives are taken through red, blue, and green glass screens respectively. From the negatives three collodion positives are produced, which, after development in complementary colour baths, are superimposed, the result giving the complete picture. The time of exposure is from 3 to 30 seconds.

Prof. Roberts-Austen exhibited an Apparatus to Illustrate M. Daniel Berthelot's Interference Method of Measuring High Temperatures. One of the beams of light in an interference apparatus traverses a heated porcelain tube, and the other beam traverses a tube of equal length containing rarefied air. When interference takes place it indicates that the air in the two tubes is equally rarefied, and therefore the temperature of the heated tube can be calculated from the pressure of the air in the other tube.

But perhaps the most interesting and important of all the exhibits was Prof. Oliver Lodge's demonstration of Recording Telegraphic Signals through Space by means of the Hertz Wave, the receiving apparatus being a Muirhead alyphon recorder.

He also shows some Apparatus illustrating the Improvements in Magnetic Space Telegraphy. The discharge of a condenser or Leyden jar round a large wire coil sets up an alternating magnetic field, which excites induced currents in another distant condenser-circuit tuned to the same frequency, causing the second Leyden jar either to overflow into a coherer or to disturb a Rutherford detector or a telephone so as to give a signal. The detector shown is a special series of small free coils and granular microphones, each coil in a permanent magnetic field, and so connected to the microphone of the next that a very feeble alternating current in the first of the series is able to make a telephone in the last emit a loud sound, or through a Langdon-Davies relay, to ring an electric bell and work a Morse sounder. A tone-telephone is also shown, which acts as a highly synchronised "call."

The Hon. C. A. Parsons had some photographs and models of his High Speed Engine, as used on the *Turbinia*. A small model of his propeller was working at about 1000 revolutions per minute.

A curious lot of crabs were shown by the Marine Biological Association. They were taken principally from the neighbourhood of Plymouth, and illustrate the remarkable faculty of self-protective concealment.

Sir David Salomons exhibited his Pseudoscope for obtaining stereoscopic effects by means of a single picture.

At 10.45 an interesting discourse was given by Sir Norman Lockyer on the Recent Solar Eclipse, as observed at Viziadrag. It was illustrated by excellent photographs.

ROYAL INSTITUTION.

General Monthly Meeting, May 9th, 1898.

Sir JAMES CRICHTON-BROWNE, M.D., F.R.S., Treasurer and Vice-President, in the Chair.

THE following were elected Members:—Messrs. Hugh Bell, H. M. Brunel, B. Knight, L. Phillips, and A. M. Smale.

The following Vice-Presidents for the ensuing year were announced:—Sir William Crookes, Sir Edward Frankland, Sir William Huggins, Dr. Ludwig Mond, the Hon. Sir James Stirling, Sir H. Thompson, Sir James Crichton-Browne (Treasurer), and Sir Frederick Bramwell (Hon. Secretary).

The Right Hon. Lord Rayleigh was re-elected Professor of Natural Philosophy in the Royal Institution.

NOTICES OF BOOKS

The Workmen's Compensation Act, 1897 (60 and 61 Vict., c. 37). With Copious Notes. By W. ADDINGTON WILLIS, LL.B. (Lond.). Third Edition. London: Butterworth and Co.; and Shaw and Sons. 1898. Pp. 96.

THE necessity for such a book as this is clearly shown by the rapidity with which the editions are following each other. There is not much comment to be made on this latest edition beyond the fact that it has been thoroughly revised, and some further notes added to the section relating to Schemes which may be submitted to the approval of the Chief Registrar of Friendly Societies, to be substituted for the provisions of the A&S. It is a very serious matter for the large employers of labour, and we hope the Frankenstein which successive Governments have been building up and fostering for the last fifty years will not turn out to be too strong for them to exorcise. There have been hopeful signs of improvement recently, but it is a thankless task to be a "master."

Mr. Willis's book is eminently practical, and it is easy to see that it is appreciated.

General Elementary Science. Edited by WM. BRIGGS, M.A., F.C.S., F.R.A.S. London: W. B. Clive, University Correspondence College Press. Pp. 140.

NOW that the University of London has made General Elementary Science a compulsory subject for the Matriculation examination, and so long as we must have examinations, a book such as the one before us—which has been compiled with a special view to this particular one—cannot fail to be of use to the unhappy examinee.

A noteworthy feature of this volume is that, comprising as it does several subjects, it is not all written by one man. Section I., on Mechanics, has been prepared by Prof. Bryan, D.Sc., F.R.S., and A. G. Cracknell, M.A.; Section II., on Heat, Light, and Electricity, by John Don, M.A., B.Sc.; and Section III., on Chemistry, by G. H. Bailey, D.Sc. Ph.D., and Frank Beddow, D.Sc., Ph.D.

The volume being, as has been shown, divided into three Sections, each Section is subdivided into a number of short numbered paragraphs, each with a special heading, and dealing simply with the subject of that heading.

After a certain number of paragraphs we find a set of questions and problems, the answers of which are to be found at the end of each Section.

The whole arrangement appears to us to be satisfactory, and likely to be of considerable service to students preparing for the examination in question. The book is well got up, and contains numerous illustrations.

The Gas Engineer's Pocket Book. By H. O'CONNOR, A.M.I.C.E. London: Crosby Lockwood and Son. 1898. Pp. 438.

"THE Gas Engineer's Pocket Book" comprises tables, notes, and memoranda relating to the manufacture, distribution, and use of coal gas, and the construction of gas works, &c. It is founded principally on copious notes taken and prepared by the author, during his daily work for many years past: they were at first intended for personal use only; fortunately, however, for gas engineers in general, he has arranged them in their present form and offered them to the world as a pocket book of reference.

This volume differs from many similar pocket books, in that it is of larger size than most of them, and not only contains the same old familiar sets of tables, but also a large amount of letterpress, describing, in well-chosen language, the different processes used in connection with gas-making, &c. It is well printed and bound, and will make a useful addition to the book-shelf.

First Year's Course of Experimental Work in Chemistry. By E. H. COOK, D.Sc. (Lond.), F.I.C. London and New York: Edward Arnold.

We cordially agree with the first sentence in the author's preface, viz., "To be successful in passing an examination is one thing, perhaps a good thing, but to have a sound knowledge of the subject is another and far more important matter." Students not only of chemistry, but of all other subjects, cannot do better than get this maxim firmly fixed in their minds. Examinations are a crude but popular method of sorting students, and too often the one who can "cram" well takes undue honours from the real plodding students who retain what they may take a longer time in learning.

Mr. Cook's book is well adapted to giving the student a good grounding in physical as well as chemical science,—in fact, it is as much a book of manipulations as of chemical facts and theories, and in the final chapter the principal laws which govern the experiments described in the preceding ones are carefully summarised. The book is likely to be of great use to beginners.

An Elementary Course of Practical Organic Chemistry. By F. C. GARRETT, M.Sc. (Vi&. et Dunelm), and A. HARDEN, M.Sc. (Vi&.), Ph.D. London, New York, and Bombay: Longmans, Green, and Co. Pp. 72. 1897.

THE small volume we have just looked through can only be considered as giving the merest outline of organic chemistry; in fact, the authors state that care has been taken to describe only such experiments as can be successfully carried out in the somewhat limited periods of time which are at the disposal of students in technical schools and colleges. If the student who goes through this primer will remember that he has still a great deal to learn, all may be well with him, and the instruction herein gained will make a good foundation for more serious work to follow.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 14, April 4, 1898.

Electric Conductivity of Solutions of Permanganate of Potash.—Emmanuel Legrand.—The author used Kohlrausch's method, with a telephone and an alternating current. The experiments made were on solutions containing 1/16, 1/32, 1/64, 1/128, 1/256, and 1/1024 of a molecule of permanganate of potash per litre; the values of the molecular conductivities observed varied from—

		Conductivity.	
		Observed.	Molecular.
1/16 normal	25° C.	0.00716	0.1145 10 ³
	35	0.00869	0.1390
	45	0.00992	0.1587
		to	
1/1024 normal	25	0.00012	0.1228
	35	0.00015	0.1536
	45	0.00015	0.1536

The table (a part only of which is here given) shows that at the same temperature the molecular conductivity increases with the dilution, and that when the temperature is high the conductivity increases the less rapidly as the temperature is increased.

Comparison of the Values of the Atomic Weights of Hydrogen, Nitrogen, and Carbon deduced from Physical Data with the Values deduced from Chemical Analysis.—D. Berthelot.—The molecular volume of a gas at 0° at the atmospheric pressure being equal to 1 for a gas following Mariotte's law exactly, this volume has the value $1 - \alpha$ for a gas which does not follow it. The molecular volume of hydrogen which is less compressible at 0° than the law requires is thus higher than 1; that of the other gases which are more compressible is lower than 1. The atomic weights of the four gases, calculated according to the author's formula, are—H = 1.0074, N = 14.007, C = 12.007, O = 16, with a $\pm$ error of 1/5000th on these figures.

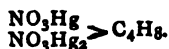
Isoquinoline and Tetrahydroisoquinoline.—Marcel Delépine.—The union of the benzenic nucleus with the pyridic nucleus to form the base C_9H_7N can be effected in two ways and gives rise to two isomers—quinoline and isoquinoline. Isoquinoline is fusible at 23° and boils at 243° without changing. Its molecular heat of combustion is—At constant volume, 1123 cal.; at constant pressure, 1123.7 cal. From which it follows that the heat of formation is $C_9 + H_7 + N = C_9H_7N$ (solid) - 35.5 cal. Hydroisoquinoline, $C_9H_{11}N$, is a liquid boiling at 234°, attracting carbonic acid from the air, like benzylamine. Its heat of combustion at constant volume = 1213.75 cal., and at constant pressure = 1215.0 cal.; therefore its heat of formation is $C_9 + H_{11} + N = C_9H_{11}N$ (liquid) + 13.2 cal. The chlorhydrates only have been studied. The displacement of isoquinoline in its chlorhydrate by potash gave at 20.8 cal.: if isoquinoline was displaced in the anhydrous state we should get $13.6 - 6.35 = 7.25$ cal. This clearly shows that the base is displaced in the form of hydrate. The calculated value would be: $13.6 - 5.41 = 8.2$ cal.

Estimation of Small Quantities of Carbonic Oxide in the Air and in Normal Blood.—L. de Saint-Martin.—A reply to a recent paper by M. A. Gantier, who had cast doubts on the extreme accuracy of the present author's method of estimation.

On the Spectrum and the Nature of Neodymium.—Eugene Demarcay.—(See p. 219).

Action of Oxidants on some Nitrified Bodies.—Oechser de Coninck.—The author gives the results of his experiments on the oxidation of some of the nitrified functions of the amines. Hydroxylamine is instantaneously decomposed, with disengagement of nitrogen, by solutions of hypochlorites of Ca, Na, K in an excess alkali. Acetaldoxime is easily decomposed in excess of the base. The diamines do not give off much nitrogen, but ammonia is separated. Carbonate of *guanidine* is immediately decomposed by the hypochlorites. It makes a good lecture experiment. The solution first takes a deep yellow colour, then an orange-red. The reaction is very distinct with the hydrazines; nitrogen commences to come off at once and continues to do so until cold. Cyanic and cyanuric acids are immediately destroyed by hypochlorites, but the results with most of the alkaloids, such as piperidine, nicotine, cocaine, &c., were negative; with antipyrine, on the contrary, a certain quantity of nitrogen was given off.

A Combination obtained with Nitrate of Mercury and Trimethylcarbinol.—G. Denigès.—In endeavouring to find out whether the tertiary alcohols, which resemble the phenols in so many ways, would furnish compounds with mercury of the same order as the latter, the author finally succeeded in isolating a compound obtained by the action of mercuric nitrate and trimethylcarbinol, but he was unable to prepare corresponding compounds with the other tertiary alcohols. He was, however, more fortunate with mercuric sulphate, which in a strongly acid solution proved to be a much more general reagent, especially with the ethylenic carbides. Mercurio-mercuric-dimethyl-ethylenic nitrate, which was formed, detonates by a shock, or on the temperature being raised to 80°; treated with HCl it effervesces like a carbonate, and gives off dissymmetrical dimethylethylene, leaving a residue, formed of mercuric and mercurous chlorides. The compound is not attacked in the cold by caustic alkalis, but after boiling for a few minutes it gives a brown precipitate, a mixture of mercuric and mercurous oxides. These different reactions show that it contains mercury in two states (at the maximum and the minimum). Its formula is—



Berichte der Deutschen Chemischen Gesellschaft.
1898, No. 3.

Gasometric Apparatus.—Otto Bleier.—The author describes an apparatus by which great accuracy in measuring gases can be obtained. A double pipette, in which the gas is measured, is connected with a compensator, through a differential manometer. Readings can be taken accurately to 0.001 per cent.

Action of Hydroxylamines on Imidchlorides and Anilidoximes.—H. Ley.—Hydroxylamine was dissolved in absolute alcohol and decomposed with the calculated amount of sodium ethylate. After rapid washing from common salt, the solution was decomposed with a solution of benzanilidimid chloride in absolute ether and left to stand for some hours. After distillation of the solvent the anilidoxime was extracted with water and re-crystallised from alcohol (m. p. 138°). Benzenyl-*o*-toluidoxime, benzenyl-*p*-chloranilidoxime, and benzenyl-*o*-nitranilidoxime were also prepared, and the action of imidchloride on α and β substituted hydroxylamines was investigated.

The End-product of the Action of Nitrogen Chloride on Dimethylaniline.—W. Hentschel.—The author studies the decomposition products of nitrogen chloride on dimethylaniline in different solutions. An alcoholic sodium solution gives tetrachloraniline (m. p. 88°). By boiling a 20 per cent solution in absolute alcohol for several hours, besides methylamidhydrochloride, he succeeds in isolating the symmetric trichlorophenol. With nascent hydrogen it yields trichlormethylaniline.

Synthesis of Naphthindol Derivatives.—O. Hinsberg and A. Simcoff.—By boiling aromatic primary or secondary amines with glyoxal-sodium bisulphite in aqueous dilute alcoholic solution, either derivatives of glycolcol or indosulphurous acid are obtained. In the latter way react α and β naphthylamines, monoalkylnaphthylamines, and some monoalkylanilines. From the products of such reactions the authors prepare various naphthindol derivatives.

Chemical Nature of Diastase.—Thomas B. Osborne.—The author justifies his own statements made in a former paper, which were contradicted by Wroblewski. His own preparation of diastase was much stronger than Wroblewski's in its inverting power on starch, and when heated to 50° or 60° did coagulate. Also it cannot contain any arabine, because it gives the characteristic test for albumin, and especially the biuret reaction, which would be obscured by the presence of a carbohydrate.

Products of the Action of Formaldehyd on Gallic Acid.—Richard Möhlau and Leopold Kahl.—The authors obtained four products by the action of formaldehyd on gallic acid. Two of these were crystalline, one soluble and the other almost insoluble, and two amorphous, one soluble and one insoluble, methylendigallic acids.

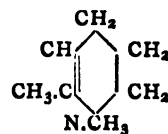
Formaldehydtrioxyfluorondicarboxylic Acid.—R. Möhlau and L. Kahl.—The authors prepare this substance from the slightly soluble crystalline methylendigallic acid. The latter is dissolved in strong H_2SO_4 and warmed gently on the water-bath to about 50°; the solution is first green, then intensely blue. Directly the blue colour appears it is cooled, and the calculated quantity of nitrosylsulphuric acid added. The reaction begins at once, but the mixture must stand for twenty-four hours before it is complete, when the dye can be extracted. It is a violet crystalline powder. Various salts of the acid are also prepared.

Occurrence of Choline and Trigonelline in the Seeds of Strophanthus and the Preparation of Strophanthine.—Hermann Thoms.—The author finds that the two bases choline and trigonelline, are found in the seeds of *Strophanthus*, beside strophanthine.

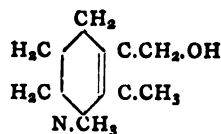
α -Methylpyrrolone, N-methyl- α -methylpyrrolone, and N-methyl- α -methylpyrrolidine.—R. Heilscher.

Condensation Products of Furfurols and Furfuracroleins.—H. Röhmer.—Furfurol was condensed by the two acids, pyroracemic and phenylacetic acid, and the products examined; also a new series of condensation products of furfuracroleins prepared.

Isomers in the Piperidine Series.—A. Ladenburg.—The author gives to the substance which is obtained by the action of formic aldehyd on—



the formula—



By adding on 2 atoms of H, and subsequent removal of a molecule of H_2O , it forms N-methyl- α - β -ethylenpiperidine.

α -Ethylpiperidine and its Methyl Derivative.—A. Ladenburg.—These substances were prepared in a pure state, free from γ -ethylpyridine, by fractional distillation and re-crystallisation of the mercury salt, which boils at 147–150°.

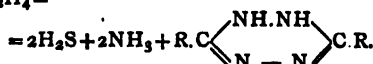
N-methylpipercoline.—A. Ladenburg.—By reduction of the above substances with sodium and alcohol, N-methylpipercoline is formed, which was prepared, and some of its salts.

Action of Diazomethane on Aromatic Nitroso-Bases.—H. v. Pechmann and Wilhelm Schmitz.—The authors investigated the behaviour of diazobenzoyl towards aromatic nitroso-bases. First nitrosomethylaniline, and then nitrosoaniline, nitrosodiethylaniline, and *m*-oxy-nitrosodiethylaniline. The experiments showed that the reaction was similar to that between diazomethane and nitrosobenzol, glyoxim-N-phenyl ethers being formed, distinguished by their red colour and basic reaction.

Relations between Safranines and Isorosindulines and Rosindulines.—Otto Fischer and Eduard Hepp.

Action of Diazomethane and of Methyl Iodide and Potash on Nitrosophenol.—H. v. Pechmann and Eugen Seel.—Nitrosophenol and diazomethane react on one another with evolution of N₂, and from the reaction-products two substances can be isolated—chinonoxim-methyl ether and paradioxyglyoxim-N-phenyl ether.

New Method of Preparing Tetrazin Derivatives.—A. Junghahn.—The author has prepared the derivatives of tetrazin by the action of hydrazin on the thiamides, the reaction being represented by the equation—



Azimido Compounds of Benzimidazole.—St. von Niementowski.—By the action of HNO₃ on the *o*-amido-benzimidazoles, new compounds are formed which are true diazo-compounds. They form a homologous series of the general formula C₇H_{2n-12}N₄. A number of these were prepared and their properties investigated.

A Fluorescent Substance.—Br. Pawlewski.—When benzylchloride reacts with resorcin, the mixture being warmed on a water-bath, a fluorescent substance is formed. A red oily mass separates out, which, after many times extracting with boiling water and treating with aqueous alcohol, gives a red amorphous powder, which melts at 315–320°, and is difficult to analyse. It appears to have the formula C₂₆H₁₈O₄. It is not very soluble, and its alcoholic solution shows a green fluorescence, while an alkaline solution shows a red fluorescence, which on dilution becomes bright green.

MEETINGS FOR THE WEEK.

MONDAY, 16th.—Society of Arts, 8. (Cantor Lectures). "Electric Traction," by Prof. Carus Wilson.

TUESDAY, 17th.—Royal Institution, 3. "The Historical Development of Modern Europe," by Samuel Rawson Gardiner, M.A., D.C.L., LL.D.

WEDNESDAY, 18th.—Society of Arts, 8. "The Evolution of the Cycle," by J. K. Starley.
Microscopical, 7.30. Exhibition of Microscopic Aquatic Life.

THURSDAY, 19th.—Chemical, 8. "The Action of Formaldehyd on Amines of the Naphthalene Series," by G. T. Morgan, B.Sc. "On the Constitution of Oleic Acid and its Derivatives" (Part I.), by F. G. Edmed, B.Sc.
Royal Institution, 3. "Heat," by The Right Hon. Lord Rayleigh, F.R.S., &c.
Society of Arts, 4.30. "Chartered Companies and Colonisation," by Sir Alfred Comyns Lyall, G.C.I.E., K.C.B., D.C.L., Member of the Council of India.

FRIDAY, 20th.—Royal Institution, 9. "The Early Life and Work of Shakespeare," by the Rt. Hon. D. H. Madden, LL.D.

SATURDAY, 21st.—Royal Institution, 3. "The Biology of Spring," by J. Arthur Thomson, M.A.

MASON UNIVERSITY COLLEGE, BIRMINGHAM.

FACULTY OF SCIENCE.

RESEARCH SCHOLARSHIPS.

(Founded by the late T. Aubrey Bowen, Esq., of Melbourne, Australia).

- (a) Two BOWEN SCHOLARSHIPS in ENGINEERING, each of the value of about £96.
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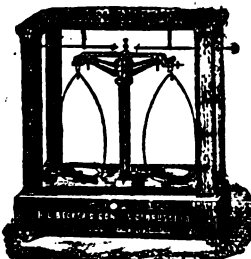
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No. 55. Fig. 7. STUDENT BALANCE, in french polished mahogany glass case, with counterpoised front sliding frame, steel knife edges, and needle pointer, with nickel pans, to carry 30 grammes in each pan and turn to half a milligramme, but with flat pans £2 9 0



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Fig. 11. No. 91.

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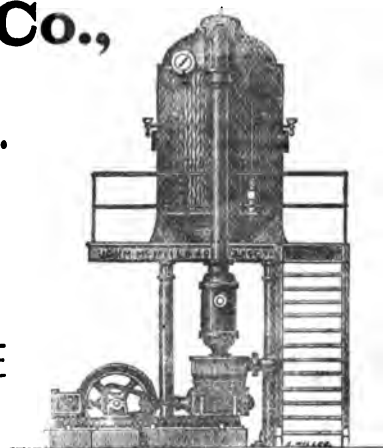
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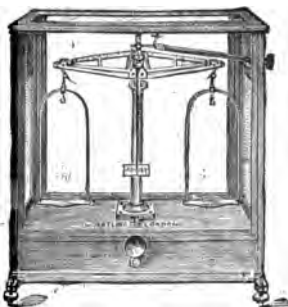
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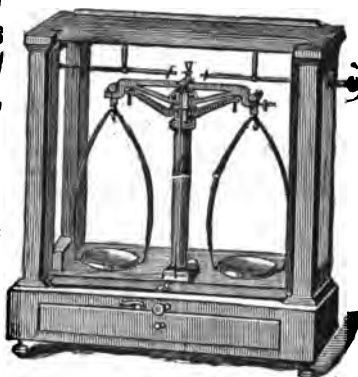


FIG. 3a.

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THE CHEMICAL NEWS.

VOL. LXXVII., No. 2008.

LIQUID HYDROGEN.

At the meeting of the Royal Society on the 12th inst. Professor Dewar contributed a preliminary note on the liquefaction of hydrogen and helium.

Professor Dewar said that in 1895 he described apparatus for the production of a jet of hydrogen containing liquid, and showed how such a jet could be used to cool bodies below the temperature that could be reached with liquid air, though all attempts to collect the liquid hydrogen failed. So far, no investigator had improved on the results described in 1895, and, as the type of apparatus employed in those experiments worked well, it was resolved to construct a much larger liquid air plant, and to combine with it circuits and arrangements for the liquefaction of hydrogen. The apparatus took a year to build up, and many months were occupied in testing and in making preliminary trials.

The many failures and defeats need not be detailed. On May 10 an experiment was started with hydrogen cooled to $-205^{\circ}\text{C}.$, and escaping continuously under a pressure of 180 atmospheres from the nozzle of a coil of pipe at the rate of 10 to 15 cubic feet a minute, in a vacuum vessel doubly silvered, and of special construction, surrounded with a space kept below $-200^{\circ}\text{C}.$ With these arrangements liquid hydrogen began to drop from this vacuum vessel into another, doubly isolated by being enclosed within a third, and in five minutes 20 c.c. of liquid were collected. The hydrogen jet then froze up from the solidification of air in the pipes of the apparatus. The yield of liquid was about 1 per cent of the gas. In the liquid condition the hydrogen was clear and colourless, showing no absorption-spectrum, and the meniscus was as well-defined as in the case of liquid air. The liquid must have a relatively high refractive index and dispersion, and the density must be in excess of the theoretical values, viz., 0.18 to 0.12 , deduced respectively from the atomic volume of organic compounds and the limiting density found by Amagat for hydrogen gas under infinite compression. Professor Dewar's old experiments on the density of hydrogen in palladium gave a value for the combined body of 0.62 , and it would be interesting to find the real density of the liquid substance at its boiling-point. No arrangements being at hand to determine the boiling-point, two experiments were made to prove the excessively low temperature of the boiling fluid. In the first place, when a long piece of glass tubing, sealed at one end and open to the air at the other, was cooled by immersing the closed end in the liquid hydrogen, the tube immediately filled, where it was cooled, with solid air.

The second experiment was with a tube containing helium.

The Cracow Academy *Bulletin* for 1896 contained a paper by Professor Olzewski entitled a "Research on the Liquefaction of Helium," in which he stated that, as far as his experiments went, helium remained a permanent gas, and apparently was much more difficult to liquefy than hydrogen. Professor Dewar, however, suggested that hydrogen and helium would probably be found to have about the same volatility, as was the case with oxygen and fluorine. Having a specimen of purified helium, extracted from Bath gas, sealed up in a bulb with a narrow tube attached, he placed the latter in the liquid hydrogen, whereupon a distinct liquid was seen to condense. From this result it would appear that there could not be any great difference in the boiling-points of hydrogen and helium.

In conclusion, Professor Dewar pointed out that all known gases had now been condensed into liquids which could be manipulated at their boiling-points under atmospheric pressure in suitably arranged vacuum vessels, though even so great a man as Clerk-Maxwell had doubts as to the possibility of ever liquefying hydrogen. With liquid hydrogen as the cooling agent, a temperature could be reached within 20° or 30° of the zero of absolute temperature, and its use would open up an entirely new field of scientific inquiry. No one could predict the properties of matter near that zero. Faraday liquefied chlorine in the year 1823. Sixty years afterwards, Wroblewski and Olzewski produced liquid air, and now, after an interval of fifteen years, the remaining gases—hydrogen and helium—were obtained as static liquids. Considering the step from the liquefaction of air to that of hydrogen was relatively as great in a thermo-dynamic sense as that from liquid chlorine to liquid air, the fact that the former had been achieved in one-fourth the time needed to accomplish the latter proved the greatly accelerated rate of scientific progress in the present age.

The paper ended with an acknowledgment of the aid rendered by Mr. Robert Lennox, without whose engineering skill, manipulative ability, and loyal perseverance the present successful issue might have been indefinitely delayed.

ON THE METALLIC PHOSPHIDES.

By A. GRANGER.

M. ALBERT GRANGER has recently published an important research on the metallic phosphides. After having made a critical examination of the attempts of his predecessors, he reviewed successively the different methods used for the preparation of compounds of metals with phosphorus; he then describes a certain number of these compounds. We here give the principal results obtained by him.

Preparation of Phosphides.

Action of Phosphorus on Metals.—By using the arrangement first designed by Schrötter, that is to say, by heating the metal under experiment, in a current of vapour of phosphorus carried by an inert gas, M. Granger was able to prepare a certain number of metallic phosphides; he also noted that some metals remained unattacked by phosphorus within a fairly wide range of temperature. The author thought it advisable to repeat nearly all the work of his predecessor, as much because of the uncertainty surrounding his results as on account of the meagreness of details contained in his memoir. It is plainly shown that if many of the compounds are disputable it is for two reasons only:—

1. A part of the metals submitted to experiment were not in a sufficiently pure state.

2. By not taking the precaution of cooling his apparatus rapidly in the vapour of phosphorus, the Viennese *savant* partially decomposed his phosphides. In fact, the current of gas at a still sufficiently low temperature might cause a loss of phosphorus, since the tension of dissociation could not be established, the atmosphere of the apparatus being unlimited. By taking the precaution of cooling the apparatus as rapidly as possible, and continuing the circulation of the current of phosphorus vapour over the still warm material, this loss of phosphorus can be avoided and pure crystalline phosphides can be obtained.

With metals, phosphorus forms bodies which may be considered as alloys of phosphorus and the metal, and which, by their peculiar properties, have been sometimes taken for definite compounds. Some of them even, by a species of pseudomorphism, are able to take not only the appearance of but even a crystalline form, which at first sight confounds them with other distinct compounds having a different composition.

Under the same conditions of temperature we always

find similar proportions of phosphorus; but in repeating the experiment, even very closely, we can always find small variations which are not met with in actual phosphides. Among these alloys we may mention Cu_3P and Fe_4P , first described by Abel and then by Sidot. While insisting on the action of phosphorus on these two bodies, we see that the quantity of the metalloid retained by the metal augments with the duration of the heating, and that it tends towards a limit established by a definite compound, which may be reached if the experiment be sufficiently prolonged. Thus the phosphide approaching Cu_3P at the temperature of formation can be transformed into a crystallised compound, Cu_3P_2 , perfectly distinct, both in its physical and chemical properties, from alloys more or less resembling Cu_3P . By keeping subphosphide of iron in a melted state, phosphorus is given off, and M. Sidot believes that this shows not a decomposition but a purification of the body, by thus getting rid of the excess of phosphorus. By prolonging the time of heating, more or less, we obtain products more or less dephosphorised, and, amongst others, Fe_4P .

Before the time of Sidot another phosphide had already been described, Fe_3P . Freese acknowledges this in an extended work on phosphides of iron (*Pog. Ann.*, cxxxiii., 225), a work in which he shows that these much too numerous bodies are, for the most part, only mixtures.

Action of Phosphoretted Hydrogen on Metals.—This method of working, though adopted by several chemists, offers no advantage. At the temperatures to which one is obliged to carry the metals to effect the combination, sufficient phosphorus is set at liberty by the decomposition of the gas to make the operation practically the same as the former one, since we are only heating the metal in a vapour of phosphorus carried by an inert gas.

Action of Metals on Phosphoric Acid and the Phosphates, either alone or in the presence of Carbon.—Some experimenters have tried in vain to prepare phosphides by heating an oxidisable metal with phosphoric acid or a phosphate. By reason of the high temperature necessary to produce the reaction only bodies very poor in phosphorus can be obtained. By the addition of carbon, phosphorus is set at liberty, and is able to combine with the metallic element; but as it is still necessary to have recourse to a high temperature for a considerable time (which is a great drawback), we can only obtain satisfactory results in the very rare case of the phosphide being stable at a high temperature.

Reduction of Phosphates or Phosphites by Carbon or Hydrogen.—The reduction of phosphates and phosphites gives nothing but altered products. As it is necessary to operate at a high temperature, and to prolong the action of the reducing agent, the conditions are such as to render the decomposition of the phosphides at the moment of their formation a matter of great simplicity. It is, in fact, impossible to maintain an atmosphere saturated with the vapour of phosphorus, so that dissociation is extremely easy.

Action of Phosphoretted Hydrogen on some Metallic Compounds.—*A priori* it seems possible to isolate phosphides by passing phosphide of hydrogen over bodies such as chlorides, oxides, or sulphides of the metals. In practice it is of advantage to substitute the vapour of phosphorus for the phosphoretted hydrogen, when precisely the same reactions take place.

Action of Phosphoretted Hydrogen on Metallic Solutions.—There is complete disaccord between the results obtained by different authors, but by repeating their experiments these discrepancies can be easily accounted for. Here several phenomena take place simultaneously; first of all the acid of the salt is set at liberty, and a precipitate of phosphide formed, which may be mixed with phosphorus, but, as the production of acid increases, there is a secondary reaction of the acid on the metallic phosphide and on the phosphide of hydrogen. The temperature and the degree of concentration of the solution are not without influence, so that as a result several re-

actions take place simultaneously, giving different results, and we can only procure very complex mixtures.

Action of Phosphorus on Metallic Salts in the presence of Water.—Nearly all salts give, under these conditions, mixtures of the metal and of phosphides which it is impossible to separate. In one particular case, however, by keeping phosphorus and phosphide of copper under water, pure crystallised cuprous phosphide could be seen forming.

Action of some Compounds of Phosphorus on Metals.—In face of the difficulties experienced in the experiments we have referred to, the author thought that it might be possible to isolate some phosphides by heating metals in the vapour of halogen compounds of phosphorus. With phosphorous chloride he did, in fact, obtain a chloride and a phosphide.

He has also experimented on the behaviour of the metals with the fluorides, bromides, and iodides of phosphorus. This method, however, is only applicable in the case when the combination of the metal with the halogen is soluble in a liquid without action on phosphorus.

Action of Phosphorus on some Metallic Compounds.—We can also make use of the reaction of phosphorus on the metallic chlorides for the preparation of phosphides; the phosphorus combines with the metal and eliminates the chlorine in the state of phosphorous chloride.

Action of Phosphorus and of Hydrogen on some Chlorides and Oxides.—Certain chlorides resist the action of phosphorus; but by making use of hydrogen at the same time, it has been possible to transform them into phosphides. This method of operating can also be applied to several oxides.

The Properties of the Phosphides.—Phosphides are solid bodies, generally possessed of a metallic lustre; they are all more or less decomposable by heat, so that when they are heated in a current of gas they are almost always partly decomposed. Chlorine and bromine attack them energetically, oxygen generally transforms them into phosphates. Certain phosphides—such as the sesquiphosphides of iron, nickel, and cobalt—are oxidised with difficulty, and resist the action of acids and of aqua regia.

Melted potash oxidises all the phosphides, as does also bromide potash. Mr. Granger has attempted to prepare alkaline phosphides, but he was stopped by the impossibility of separating the phosphide formed, either from the excess of phosphorus or from the excess of metal. These impure phosphides, prepared by direct action, are brownish bodies, of a waxy consistency, very inflammable, and decomposed by moisture.

Magnesium combines with phosphorus at about 600° with great ease; but at this temperature it attacks all substances in which it may be placed, so that it is impossible to isolate a pure product. Phosphide of magnesium is readily oxidisable, and decomposes on contact with water. Aluminium does not combine with phosphorus, at any rate not below 1000° .

Chromium unites directly with phosphorus at about 600° , and retains about 21 per cent, but the phosphorization is difficult to complete throughout the mass. It is a simple matter to prepare protophosphide of chromium by heating chromic chloride in a current of the vapour of phosphorus and hydrogen. The body, CrP , is of a greyish colour, and its structure is graphitoidal; it is not attacked by acids or by aqua regia.

Manganese at 700° absorbs about 24 per cent of phosphorus. But the difficulty of procuring this metal perfectly free from foreign matter prevents the isolation of pure compounds. A phosphide, Mn_3P_2 , can be prepared in thin, brilliant flakes, in the same manner as phosphide of chromium.

Iron combines easily with phosphorus. We already know three phosphides, Fe_2P , FeP , and Fe_3P_4 . By causing the trichloride to act on metallic iron, the author obtained Fe_2P_3 , and afterwards, by heating ferric chloride in the vapour of phosphorus, he transformed it into sesquiphosphide, Fe_2P_3 . These two phosphides are white in colour,

and crystallise in prismatic needles; they are not easily attacked, except by chlorine.

Nickel and cobalt, under the same conditions as iron, both form a subphosphide and a sesquiphosphide. Ni_2P exists as yellowish white crystals; Co_2P is white, in small, hard, and brilliant needle-like crystals; both are easily soluble in nitric acid. The subphosphides of nickel and cobalt resist the action of acids to the same extent as do the corresponding compounds of iron.

It was of interest to try whether with zinc, cadmium, and tin we could utilise the reaction of phosphorous chloride on the metal, or of phosphorus on the chloride, and prepare the compounds already known. The results were not satisfactory, as it was impossible to separate the products of the reaction without decomposing them.

Copper at 700° forms a subphosphide of copper; then at about 900° another phosphide, Cu_3P_2 . By heating either copper in the vapour of trichloride of phosphorus, or cuprous chloride in the vapour of phosphorus, a biphosphide of copper is formed. We have seen above how cuprous phosphide can be obtained.

Mercury does not unite directly with phosphorus. Heated with bi-iodide of phosphorus it forms a phosphide, Hg_3P_4 , crystallising in hexagonal prisms, combined with the β and α facets of a rhombohedron. Phosphide of mercury is easily decomposed under the influence of heat.

Lead, bismuth, and antimony do not combine with phosphorus directly, and all attempts to obtain such phosphides indirectly have only given amorphous bodies containing extremely small quantities of phosphorus.

Silver and chloride of silver form a biphosphide at 400° , when heated in a vapour of phosphorus. At 500° the biphosphide of silver decomposes, and gives off all its phosphorus if it is kept in a current of inert gas.

Gold behaves in the same way as silver; it combines with phosphorus at the same temperature, and gives Au_3P_4 , which is easily destroyed under the influence of heat.

Platinum is easily attacked by phosphorus; it gives extremely fusible compounds, and is very difficult to obtain crystallised. At 500° it forms a biphosphide; towards 600° a body less rich in phosphorus is formed, Pt_3P_5 ; above 700° a subphosphide is formed; and finally, at 1000° , the metal only retains a few hundredths of phosphorus.

The phosphides of platinum are attacked very slowly by aqua regia, but dissolve rapidly in melted alkalis.

Separation of Phosphoric Acid from the Metals.—To effect the separation of phosphoric acid from the metals, M. Granger has used electrolytic methods with success, in all cases where such a method was possible. Iron, nickel, zinc, cobalt, copper, cadmium, and tin, were very easily estimated, though the ordinary processes of analysis are sufficiently delicate. In nearly every case the phosphoric acid was precipitated in the form of ammonio-magnesian phosphate. In some cases the author used Chancel's reagent, and made the estimation in the form of phosphate of bismuth. The nitro-molybdic reagent has been used the least often for the separations, on account of the difficulty of freeing the liquor from the excess of molybdenum.—*Moniteur Scientifique*, Series 4, vol. xii., May, 1898.

THE ANALYSIS OF CAUSTIC SODA.

By P. DOBRINER and W. SCHRANZ.

THE analysis of caustic soda includes the estimation of the caustic alkali and of the carbonate of soda. Two methods may be used for this analysis. The alkalinity may be determined before and after the elimination of the carbonate by chloride of barium, or the total alkalinity may be determined at the same time as the carbonic acid. In a series of comparative experiments we found that, according to the first method, the proportion of carbonate of soda present appeared to be from 1 to 2 per

cent higher than the actual amount, and, naturally, the proportion of caustic soda would appear to be lower by the same quantity. We have shown that this error is due to the use of toughened folded filter-papers, for filtering the alkaline solutions treated by chloride of barium. These papers appear to absorb notable quantities of alkali. But if we use ordinary filter-papers this error disappears, and the two methods give concordant results. It is best to let the precipitated carbonate of baryta settle thoroughly, and to use a definite known quantity of the clear liquid for the determination of the caustic alkali. Results of sufficient accuracy can be obtained by the following method:—

To 2.65 grms. of caustic soda dissolved in 50 c.c. of water, add phenolphthalein, and titrate with normal sulphuric acid until the red colour disappears. Then add 3 c.c. more of the normal acid, and boil for five minutes to drive off the carbonic acid; then titrate the excess of acid by normal soda.

If a be the number of c.c. of normal acid used in the first titration, and b the number of c.c. of normal soda used in the second titration (after driving off the carbonic acid), then the proportion of caustic soda present will be $2(2a - b)$ per cent Na_2CO_3 , corresponding to NaHO and $4(b - a)$ per cent Na_2CO_3 .

It is well known that this method is based on the fact that, in the first titration, the whole of the caustic alkali is neutralised, and half the carbonate transformed into bi-carbonate—



This reaction has served as the starting-point for a large number of methods for the estimation of caustic alkalis in presence of the carbonate; but these methods only give exact results when the quantity of carbonate present is very small.

Lunge recommends titrating first with phenolphthalein as indicator, until decolouration takes place, and then to continue with methyl-orange as indicator. Those chemists who do not like the use of methyl-orange as an indicator will find the method we propose very convenient.—*Zeitsch. für Angewandte Chemie*, xiv., 455.

ON THE YTTRIC EARTHS CONTAINED IN THE MONAZITE SANDS.*

By P. SCHÜTZENBERGER and O. BOUDOUARD.

(Concluded from p. 221).

B.—THE oxides of which the atomic weights of the corresponding metals are 104.55, 104.65, and 104.2 were mixed and converted into nitrates. The method of fractional fusions gave:—

Sub-nitrate No. 1.

Atomic weight	107.95
Anhydrous sulphate used ..	2.7597
Oxide obtained	1.4453

Sub-nitrate No. 2.

Atomic weight	105.6
Anhydrous sulphate used ..	2.0655
Oxide obtained	1.0725

Sub-nitrate No. 3.

Atomic weight	102.2
Anhydrous sulphate used ..	1.3767
Oxide obtained	0.7058

Mother-liquors.

Atomic weight	103.0
Anhydrous sulphate used ..	4.8470
Oxide obtained	2.4930

* *Bull. Soc. Chim.*, Series 3, vol. xix.-xx., No. 6.

This last oxide, transformed into nitrate, was heated up to 325° and submitted to a partial decomposition :—

Sub-nitrate No. 1.

Atomic weight	101.25
Anhydrous sulphate used ..	1.0577
Oxide obtained	0.5402

Mother-liquors.

Atomic weight	102.45
Anhydrous sulphate used ..	3.4885
Oxide obtained	1.7900

C.—The oxide previously obtained, which gave 97.1, was converted into nitrate and submitted to a partial decomposition at 325°.

Sub-nitrate No. 1.

Atomic weight	104.5
Anhydrous sulphate used ..	1.1355
Oxide obtained	0.5873

Sub-nitrate No. 2.

Atomic weight	101.75
Anhydrous sulphate used ..	1.0865
Oxide obtained	0.5560

Mother-liquors.

Atomic weight	95.1
Anhydrous sulphate used ..	3.0125
Oxide obtained	1.5005

The oxides of which the atomic weights were respectively 97.45, 97.7, and 97.5 were dissolved in nitric acid. The nitrate was then partially decomposed :—

Sub-nitrate No. 1.

Atomic weight	103.8
Anhydrous sulphate used ..	1.6036
Oxide obtained	0.8271

Sub-nitrate No. 2.

Atomic weight	98.5
Anhydrous sulphate used ..	1.1945
Oxide obtained	0.6036

Sub-nitrate No. 3.

Atomic weight	95.6
Anhydrous sulphate used ..	0.8465
Oxide obtained	0.4225

Mother-liquors.

Atomic weight	96.0
Anhydrous sulphate used ..	2.5795
Oxide obtained	1.2895

D.—We united all the oxides having given 122, which represents 5.95 grms. The nitrate was submitted to partial decomposition at 325°. We obtained three sub-nitrates and an undecomposed portion, which were analysed.

Sub-nitrate No. 1.

Atomic weight	142.0
Anhydrous sulphate used ..	3.1217
Oxide obtained	1.8132

Sub-nitrate No. 2.

Atomic weight	126.0
Anhydrous sulphate used ..	2.2050
Oxide obtained	1.2250

Sub-nitrate No. 3.

Atomic weight	117.8
Anhydrous sulphate used ..	0.9655
Oxide obtained	0.5230

Mother-liquors.

Atomic weight	110.15
Anhydrous sulphate used ..	4.2024
Oxide obtained	2.2182

We now wished to find out if the oxide giving 142 was a final term. Transformed into nitrate and submitted to

a partial decomposition at 325°, it gave an insoluble sub-nitrate and an undecomposable portion.

Sub-nitrate No. 1.

Atomic weight	155.75
Anhydrous sulphate used ..	0.9855
Oxide obtained	0.5910

Mother-liquors.

Atomic weight	137.4
Anhydrous sulphate used ..	1.6968
Oxide obtained	0.9733

This last oxide, after solution in nitric acid, was decomposed afresh.

Sub-nitrate No. 1.

Atomic weight	150.2
Anhydrous sulphate used ..	0.7720
Oxide obtained	0.4570

Mother-liquors.

Atomic weight	123.25
Anhydrous sulphate used ..	0.8863
Oxide obtained	0.4884

Finally, the oxides 155.75 and 150.2 were united, transformed into nitrates, and submitted to a fresh partial decomposition.

Sub-nitrate No. 1.

Atomic weight	162.5
Anhydrous sulphate used ..	0.6425
Oxide obtained	0.3910

Mother-liquors.

Atomic weight	148.3
Anhydrous sulphate used ..	1.0500
Oxide obtained	0.6190

In face of the small quantity of material at our disposal, we were unable to continue our research, and so make quite sure that we had not reached the highest limit of decomposition. As the atomic weights of holmium, ytterbium, and erbium are in the neighbourhood of 165, we thought this should be the ultimate term. All the products having given intermediate atomic weights should therefore be considered as mixtures of these earths with high atomic weights with an earth having a considerably lower one; this latter, however, being quite distinct from yttrium. Yttrium (89.5) is not found in the monazite sands, except in very minute proportions; we have never in the course of these determinations been able to isolate more than a very small quantity of yttria (4 to 5 grms. of oxides having an atomic weight of from 93 to 94).

In spite of this great diversity in the atomic weights determined by a method so simple that an error is hardly possible, all these earths give a spark spectrum with the chloride similar to, and easily confounded with, that of the original mixture. This spectrum, above all, shows two characteristic shadowy bands to the left, formed by the juxtaposition of the lines occupying—(a) the space comprised between numbers 69 and 73 on the micrometer scale of our instrument, in which division 90 coincides with the sodium D line—

$$\lambda = 618 \text{ to } 614;$$

and (b) the space comprised between the divisions 80 and 85—

$$\lambda = 602 \text{ to } 595.5.$$

We have further noticed, but in a less marked manner, other lines, of which the most apparent are—(c) those comprised between the divisions 188 to 190—

$$\lambda = 499 \text{ to } 497.5;$$

and (d) those comprised between the divisions 217 to 220—

$$\lambda = 481.5 \text{ to } 480.$$

In conclusion, the spectra observed seem to be confounded with that of yttrium, and the conclusion to which one is led by spectrum analysis would certainly be that the earths examined by us are almost pure yttria, at

the most contaminated by only a few hundredths of foreign earths.

In a future paper I shall give the results at which I arrived by continuing the study of the yttric earths.

A REVISION OF THE ATOMIC WEIGHT OF ZIRCONIUM.*

By F. P. VENABLE.

(Concluded from p. 223).

Method of Work.

THE purified chloride was introduced into a small glass flask having a capacity of 100 c.c. This was provided with a glass stopper ground to fit, and also a second one with two tubes arranged for the passage of the hydrochloric acid gas. The arrangement of the tubes was similar to that in an ordinary ether wash bottle, though both tubes outside were bent downwards and had little bulbs blown in them for catching moisture, &c. A Thorner bath was found to be very convenient for keeping these flasks at 100° C. The hydrochloric acid was prepared by allowing sulphuric acid to drop into a large flask containing the hydrochloric acid. The gas thus obtained in a regular stream, was passed through a wash-bottle containing sulphuric acid and then through towers filled with glass beads kept moist with concentrated sulphuric acid.

The drying took from fifty to one hundred hours (in some extreme cases). If the stream of gas was rapid the temperature could rise to 110° or even higher without decomposition of the chlorine. A much lower temperature caused this decomposition if the stream was insufficient to keep the flask full of gas. A number of experiments were carried out showing these facts. Indeed, two in the series of determinations were lost by the stream of gas becoming too slow or altogether ceasing for a short while (Experiments VI. and VIII.). It was thought from experiments at first that where this decomposition had begun it was impossible to secure a constant weight of the residue, but this is certainly not true where the decomposition has been only slight. Of course, this introduces a chance for error in the method. The drying must be watched quite closely, and not more than eight or ten hours of drying could be easily managed in a day.

At first it was feared to remove the atmosphere preparatory to weighing, and efforts were made at weighing the flasks full of hydrogen chloride. These results were too low and varied among themselves, so that it was evidently impracticable to carry out the experiments in this way. It was found that the hydrochloric acid could be replaced by dry air. The flask was removed from the bath, and dry pure air passed through it for half an hour. It is, of course, essential that the air be carefully dried. The tubes are then removed and the glass stopper quickly fitted in its place. It is then ready for placing in the balance case. The chloride is deliquescent and some trouble in weighing was experienced. If the stopper was well ground there was no appreciable change in the weight in from twenty to forty minutes after placing upon the balance. The loss of weight in the latter part of the drying was very slow. The weighings were taken at intervals of from six to eight hours' heating. The number of weighings necessary before constancy was secured served as a safeguard against error. A series of such weighings is given further on.

After drying, the chloride was dissolved in a small amount of water (re-distilled) and this solution, with rinsings, transferred to a platinum crucible. It was evaporated to dryness upon a water-bath, with due precautions against dust, &c.; was next heated gradually upon a

sand-bath until most of the chlorine had been driven off, and was then slowly raised to the highest temperature attainable by the Bunsen burner. During this latter part of the operation the cover was kept on. Three or four days were thus consumed, the gradual heating giving a coherent flinty mass of glistening semi-translucency which could be safely heated by the water-blast without loss. During the driving off of the chlorine the platinum crucible was more or less attacked, but as this was before the lid was on, there was little chance of loss from this source. This corroded platinum was probably the reddish brown decomposition product mentioned by Bailey as coming from the ignition of the oxychloride.

The last of the chlorine was driven off by heating with a water-blast for from forty-five to seventy hours. The last weighings were made at intervals of from six to twenty hours, and were recorded as constant if they agreed within 0.00005 of a gram. A series of weighings is here given as an example. Experiment No. II., or the first one successfully carried out in the series, is taken for the purpose.

Weights of zirconium chloride—March 28	..	5.25910
" " " 29	..	5.25786
" " " 30	..	5.25760
" " April 1	..	5.25762

Weights of zirconium oxide—April 11	..	2.78759
" " " 12	..	2.78724
" " " 13	..	2.78583
" " " 15	..	2.78517
" " " 16	..	2.78452
" " " 17	..	2.78451

This zirconia was examined for chlorine in most of the analyses and was found to be free from it. In the last three experiments reported in the series, the ignited residue was treated with hydrofluoric acid. Ignition after this treatment was very difficult, as the mass was not compact and coherent. The finely-divided zirconia was lost in spite of the most careful treatment. The first was lost altogether and the weighing was not carried out. The second showed upon the lid signs of the powdered zirconia having been swept out. The total loss of weight, however, was less than 1 m.grm. This could readily be attributed to the zirconia and pointed to the absence of silica, unless in insignificant traces.

I think great purity may be claimed for the preparations used in the analyses. Crystallising from hot hydrochloric acid is apt to remove most known impurities, and they were eliminated beyond the possibility of detection by the ordinary tests. The analyses were not made from the same preparation, but from small quantities prepared at different times, and some crystallised ten or a dozen times more than others, yet no appreciable difference could be detected in the results. That these results are not in close accord with one another, is due in part to the deliquescence of the chloride and to the risks involved in the prolonged heating of the oxide.

Sources of Error.

Five main sources of error have occurred to me apart from any question as to the purity of the material.

1. The deliquescence of the chloride.
2. The loss of the finely-divided zirconia.
3. The corrosion of the platinum crucibles.
4. The attack of the glass flasks by the gaseous and liquid hydrochloric acid. It is very evident that the glass vessels would suffer when subjected to the action of hydrochloric acid during such prolonged periods. The weighings easily revealed the extent of this action; it was found to vary with different flasks. For instance, flask I. lost 0.00016, 0.00031, 0.00023, and 0.00078 gm. On the other hand, flask IV. weighed after the first experiment only 0.00011 gm. less, and after the next two experiments the loss was 0.00013, and the total loss during three experiments was 0.00024. This is probably trans-

* From the *Journal of the American Chemical Society*, xx., No. 2.

ferred mainly as alkaline chlorides to the crucibles, and is there volatilised. Sodium could readily be detected upon the lid of the crucible after partial ignition. Of course, if all is volatilised then no error is introduced into the experiment, but there may be some non-volatile material also transferred from the flasks. The total amount is so small that the error cannot be a large one.

5. Substances carried into the drying flasks by the stream of gas. Much care was taken to avoid error from this source. The main danger lay in the necessity for the use of some rubber connections. Vulcanised rubber was used. It was freed from excess of sulphur and was replaced by fresh pieces when attacked by the gas. The dry gas does not attack rubber very rapidly. Gaseous sulphur compounds, coming from the sulphuric acid used in preparing and drying the hydrogen chloride, were probably carried through the drying flasks, but there seems to be no probability of their causing a decomposition of the oxychloride.

The Determinations.

All analyses which were completed under the proper conditions of the method, as already given, are here reported. Experiment III. became contaminated from the iron support during the prolonged heating, and came out consequently a little high, giving the ratio of $ZrO_2 : ZrOCl_2 \cdot 3H_2O$ as 53.12. In most of the subsequent analyses the ignition was carried out with the crucible suspended in a platinum wire cage from a glass support. Experiments VI. and VIII. were dried with an insufficient stream of gas, as already stated, and hence were partially decomposed; they gave 53.57 and 53.8 respectively. The remaining analyses follow:—

	$ZrOCl_2 \cdot 3H_2O$.	ZrO_2 .	Ratio.
II. ..	5.25762	2.78450	52.961
IV. ..	5.53994	1.87550	52.981
V. ..	3.25036	1.72435	53.051
VII. ..	1.52245	0.80708	53.012
IX. ..	2.98802	1.58274	52.969
X. ..	2.11371	1.11920	52.949
XI. ..	2.38139	1.26161	52.978
XII. ..	1.90285	1.00958	53.055
XIII. ..	2.61847	1.38658	52.954
XIV. ..	1.07347	0.56840	52.951
	26.64828	14.11953	52.986

Calculating these for the ratio $ZrOCl_2 \cdot 3H_2O : ZrO_2$, taking $H=1.008$, $O=16$, and $Cl=35.45$, we have the following:—

Maximum ratio ..	53.055	Atomic weight ..	91.12
Mean ..	52.986	" ..	90.78
Minimum ..	52.951	" ..	90.61

The atomic weight, as determined by Bailey, is 90.65. The mean value given in Clarke's "Re-calculation" is 90.40. I purpose repeating the determinations with the oxychloride, with such modifications as have occurred to me since the completion of the above work.

Estimation of Phenylhydrazin.—M. Causse.—0.20 gm. of hydrochlorate of phenylhydrazin is placed in a 500 c.c. flask, and 60 c.c. of a special solution of arsenic is added. (This solution is made by adding 125 grms. of arsenic acid to 450 grms. of water and 150 grms. of HCl, filter, and make up to a litre with glacial acetic acid). The flask, fitted with a vertical condenser, is gently heated; after 40 minutes add 200 c.c. of water, neutralise with soda until distinctly alkaline to phthalein, re-acidulate with HCl, and add, when cold, 60 c.c. of bicarbonate of soda (saturated), 3 to 4 c.c. of starch water, and titrate with a decinormal solution of iodine (1 c.c. = 0.0127 I).—*Bull. Soc. Chim. de Paris*, Series 3, vol. xix.-xx., No. 4.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING APRIL 30TH, 1898.

By SIR WILLIAM CROOKES, F.R.S.,
and
PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, May 10th, 1898.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 168 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from April 1st to April 30th inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 168 samples examined by us, all were found to be clear, bright, and well filtered.

We have once more to record a deficiency in the rainfall at Oxford, though not so serious a one as last month; the actual fall during the month of April was 1.34 inches and the thirty years' average 1.66 inches, leaving a deficiency of 0.32 inch. This brings the total deficiency for the year to 3.07 inches.

Our bacteriological examinations of 237 samples have given the results recorded in the following table; we have also examined 34 other samples, from special wells, stand-pipes, &c., making a total of 271 in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	611
New River, filtered (mean of 23 samples) ..	10
Thames, unfiltered (mean of 24 samples) ..	7854
Thames water, from the clear water wells of five Thames-derived supplies (mean of 118 samples) ..	17
Ditto ditto highest	231
Ditto ditto lowest	0
River Lea, unfiltered (mean of 24 samples) ..	2070
River Lea, from the East London Company's clear water well (mean of 24 samples) ..	24

As we pointed out last month, the continued small rainfall in the Thames valley has had a very beneficial effect on the quality of the water; both the bacteriological results in the table above, and the comparative chemical results in the following table, show this in a marked manner. The quality of the water is in every respect excellent:—

Comparison of the Averages of the Five Thames-derived Supplies for the Months of April, 1897 and 1898.

Common Salt. Per gall. Means.	Nitric Acid. Per gall. Means.	Hardness. Degrees. Means.	Oxygen. reqd. Per gall. Means.	Organic Carbon. Per gall. Means.	Organic Carbon. Per gall. Max.	Colour. Br's: Blue. Means.
April, 1897. 2.030	1.020	15.99	0.043	0.097	0.129	9.0: 20
1898. 2.165	0.775	14.83	0.032	0.074	0.109	10.1: 20

We are, Sir,

Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 5th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

Messrs. E. C. Jee, W. Marshall, and T. H. Pope, were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Harry Brearley, Totley, Derbyshire; Arthur Stanley Hemmy, Government College, Lahore; Leonard Myddleton Nash, 281, Seven Sisters' Road, Finsbury Park, N.; Edward John Russell, Owens' College, Manchester; Sigmund Stein, 323, Vauxhall Road, Liverpool; Frank Edwin Weston, 43, Larkhall Rise, Clapham, S.W.; William Williamson, 86, Tyndall Road, Leyton, N.E.; Charles Arthur Wrench, 3, Parklands, Surbiton Hill.

Of the following papers those marked * were read:—

*69. "*The Action of Hydrogen Peroxide on Carbohydrates in the presence of Iron.*" By C. F. CROSS, E. J. BEVAN, and CLAUDE SMITH.

The results obtained by H. J. H. Fenton (*Trans.* 1894, lxx., 899; 1895, lxxii., 48; 1896, lxxix., 546) in oxidising tartaric acid by hydrogen peroxide in presence of soluble iron compounds suggested the application of the method to other hydroxy-compounds, and notably to the carbohydrates. The authors have studied the behaviour of typical hexoses and of cane-sugar, when treated in aqueous solution with hydrogen peroxide at ordinary temperatures, and, in confirmation of Fenton's observations, have found in this group also that the presence of iron compounds is an essential condition for the production of the characteristic reactions. A convenient proportion of iron (Fe as $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) is 1/10000 of the weight of a solution containing 4 grms. of the carbohydrate in 100 c.c. The authors reserve for the present any statement of the limiting proportion which may be necessary, and are engaged in ascertaining whether other inorganic compounds may not be found to have similar effects.

With such proportions and with hydrogen peroxide in quantities sufficient to supply 1 or 2 atoms of oxidising oxygen for each molecule of hexose, reaction takes place readily with marked rise of temperature (10 – 20°), but in the absence of iron compounds, all other conditions remaining the same, nothing happens at ordinary temperatures. After reaction the solutions are acid to the taste. The quantity of acid formed is greater from dextrose than from levulose. The volatile acids separated by distillation are formic and acetic acids, and represent 15–20 per cent and 4–7 per cent respectively of the weight of the dextrose. The non-volatile acids represent about one-half the total acidity, and contain a dicarboxylic acid which is easily isolated by precipitation as lead salt in the presence of acetic acid, and on analysis gives numbers corresponding with those required for tartronic acid.

After removal of the acids, the presence of furfuroids is identified by estimations of furfural in the distillates from hydrochloric acid (sp. gr. 1.08). The quantities obtained from dextrose or cane-sugar are 3–4 per cent, representing 7–9 per cent of the furfuroid. The products yielding furfural are not acid in character, but there is no evidence that they are pentoses. From levulose, only traces of these products are formed. The solutions give a well-marked iodoform reaction, indicating that the hexose molecules undergo internal re-arrangement, and that the phenomena are not those of a simple oxidation.

The characteristic products of the reaction are separated from solution by the addition of alcohol and ether; on drawing off the denser aqueous layer, a solution is obtained which dries in a vacuum to a gummy solid, destitute of any appearance of crystallisation. These compounds

react with phenylhydrazine acetate in the cold, forming osazones, and give evidence of the presence of highly reactive groups by reducing Fehling's solution in the cold. The yield of osazones is considerable, amounting to 30–60 per cent of the weight of the carbohydrate in the case of levulose and of cane-sugar, and to 12–20 per cent in that of dextrose. Two groups of osazones have been obtained:—(1) compounds melting at 185 – 195° , and resembling the glucosazones in properties, but differing from them in composition, the nitrogen (N=17–20 per cent) being 2–3 per cent higher; (2) compounds with a low melting-point (130°), and freely soluble in hot water. From these results it might be inferred that the products are the "osones" or "oxyglucoses" of Fischer, but they resist the action of zinc and acetic acid on the one hand, and of bromine on the other, and are not reduced by sodium amalgam in solutions kept slightly acid,—properties which differentiate them from the normal carbohydrates and from such of their oxy-derivatives (ketaldoses) as are at present known.

The investigation of the nature of these products is complicated by the fact that no crystalline derivatives other than the osazones have been obtained. No definite acetates or benzoates have been isolated. Some evidence as to their relationship to the original hexoses, however, is obtainable from a closer study of the constants of the reaction. It appears, in the first place, that there is no simple proportion between the quantity of hydrogen peroxide employed and the amount of the characteristic products obtained. Having established this for quantities representing 3, 2, and 1 atoms of oxygen for each molecule of the hexose, the authors found that very considerable effects were still produced on further reducing the proportion of peroxide. Thus from 40 grms. dextrose treated with sufficient peroxide to furnish only 1/10 atom of oxygen for each molecule of hexose, the quantity of osazones formed in the cold from the product of the reaction was 8 grms., an amount as great, therefore, as that obtained when ten times this proportion of the peroxide was employed. Similarly, the addition of still smaller quantities of the peroxide to a dextrose solution was found to convert a considerable proportion of the hexose into compounds not fermented by yeast, though reducing Fehling's solution and otherwise resembling the compounds just described.

The authors conclude (1), that hydrogen peroxide acts primarily by determining a constitutional change in the hexose molecule, *i.e.*, by internal re-arrangement, such effects bearing no direct proportion to the quantity added; and (2), that the oxidising actions observed—*s.g.*, the formation of dicarboxylic acids—are subordinate or secondary effects.

As regards the nature of the constitutional changes in question, the reactions of the characteristic products indicate the presence of $-\text{C}(\text{OH})\text{:C}(\text{OH})-$ groups. In the formation of dihydroxymaleic acid from tartaric acid, this radical results from an actual removal of hydrogen by oxidation, and in the carbohydrates might be formed by internal re-arrangement. The authors consider that such a change is the primary effect of contact with the peroxide, although $-\text{CH}(\text{OH})\text{-CH}(\text{OH})-$ residues also are probably attacked and hydrogen eliminated by direct oxidation.

The obvious bearings of these results upon the problem of plant physiology, from which point of view they are positive and sufficiently established, induce the authors to put forward this preliminary communication without waiting for a definite solution of the constitutional problems, which are still under investigation.

DISCUSSION.

Dr. ARMSTRONG referred to the interest of the work in connection with vegetable physiology, indicative as it was of the far-reaching character of the interactions concerned in the elaboration of the products of plant activity. Although the part played by enzymes in such processes

had been generally recognised, their importance must be much accentuated now that Buchner's remarkable discovery of an enzyme capable of inciting alcoholic fermentation was placed beyond doubt. It seemed quite possible, from the experiments of Mr. Cross and his colleagues, that iron, which is known to be present in most plants, may behave to some extent as an enzyme, and thus possess unexpected importance from the point of view of plant chemistry; it was very noteworthy from this point of view that the formation of reduction products yielding iodoform had been observed as well as that of oxidation products.

Dr. MORRELL stated that, with galactose, the disappearance of hydrogen peroxide is twice as rapid as with glucose, and that the addition of large quantities of the peroxide caused a marked rise of temperature. Ferrous iron, which could not be detected during the oxidation, reappeared when hydrogen peroxide could be recognised no longer in the solution.

Mr. FENTON said that, since he had observed the peculiar influence of iron in the oxidation of tartaric acid, experiments had been made in other directions, and as so wide a field had been opened up for investigation the co-operation of the authors and of Dr. Morrell was most welcome.

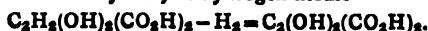
Mr. LING, referring to the possible analogy between the products of the reaction described by the authors and those formed by the oxidation of sugars by alkaline copper solutions, said that Kjeldahl had observed that glucose and fructose, when boiled with a solution of potassium copper sulphate (Ost's solution), yield mesoxalic acid. Kjeldahl had also shown that this acid, on treatment with phenylhydrazine acetate, is converted into a dihydrazide melting at 148° —a derivative, therefore, of dihydroxymaleonic acid. Might not the supposed osazone, which was formed in the cold, be a substance of this kind?

Mr. CROSS, in reply, stated that the phenylhydrazine derivatives obtained in the cold had the characteristics of osazones, although neither aniline nor ammonia, which, according to Fischer, are simultaneously produced in the osazone reaction, could be detected in the filtrates. It had not escaped attention that the derivatives might possibly be hydrazides resulting from condensation with a $-C(OH):C(OH)-$ group, and they were being investigated from this point of view.

*70. "Note on the Oxidation of certain Acids in presence of Iron." By HENRY J. HORSTMAN FENTON, M.A.

It has been shown in several previous communications (*Trans.*, 1894, lvi., 899; *B. A. Report*, 1895, &c.) that when tartaric acid is oxidised in presence of a small quantity of ferrous iron, 1 molecule of the acid loses 2 atoms of hydrogen, giving rise to dihydroxymaleic acid. The most effective oxidising agent for the purpose is hydrogen dioxide, but the result is also brought about by chlorine, hypochlorites, bromine, &c., and by atmospheric oxygen in presence of sunlight. The presence of ferrous iron is essential, but its proportion seems to bear but little relation to the yield of acid in the ordinary course of preparation, the action being, in fact, what is usually termed catalytic. It is necessary that the addition of iron shall precede that of the oxidising agent. From the fact that dihydroxymaleic acid, on heating with water, yields glycollic aldehyde, and that this readily condenses to a hexose, it is evident that the change may afford information with regard to the natural formation of carbohydrates.

The production of dihydroxymaleic acid from tartaric acid is most easily represented by assuming the removal of the two non-hydroxylic hydrogen atoms—



It is true that dextrotartaric acid would be expected, in this manner, to yield a fumaroid instead of a maleoid acid, but it is quite possible that "stereomeric" change takes place during one of the stages of preparation, and in any case this objection can hardly be considered a

serious one, bearing in mind the large number of known exceptions (Michael, *J. Prakt. Chem.*, 1892, xli., 400).

While seeking for a chemical explanation of the part played by the iron, the following may be offered as a provisional suggestion. The two non-hydroxylic hydrogen atoms in tartaric acid may be supposed to possess feebly acid functions owing to the neighbourhood of the CO_2H- and $OH-$ groups, and it is possible that an atom of divalent iron may replace these two hydrogen atoms, giving the compound—



On addition of the oxidising agent the iron atom assumes the trivalent state, and can therefore no longer be "retained." The result is an unsaturated acid, and the iron enters into solution as a ferric salt, e.g., ferric tartrate. Dihydroxymaleic acid readily reduces ferric salts in the cold, experiment indicating that 2 atoms of iron are reduced by 1 molecule of the acid, so that the ferrous iron is regenerated at the expense of a portion of the acid.

A large number of the commoner acids have been investigated as regards their behaviour in this respect. In addition to all the non-hydroxylic acids examined, lactic, malic, citric, and tartronic acids give negative results. If, however, the above explanation be accepted, it is evident that the production of the violet colouration would only be expected in the case of those acids the molecules of which contain at least two $CH(OH)-$ groups. Mucic and saccharic acids might therefore be expected to yield positive results, and recent experiments have shown that this is the case. Both these acids, when treated in the cold with a limited quantity of hydrogen dioxide in presence of ferrous iron, give intense violet colourations. Attempts are being made to isolate the oxidation products, which appear to be even less stable, however, than dihydroxymaleic acid.

DISCUSSION.

Dr. ARMSTRONG thought a more probable explanation of the part played by iron to be that the iron salt became added to the carboxylic group; if changes then occurred such as he had suggested take place when optical inversion is effected by hydrolytic agents (*Trans.*, 1896, lxi., 1399), it would be easy to understand why dihydroxymaleic acid was obtained instead of the fumaric derivative.

Mr. FENTON, referring to the suggestion he had made, said that replacement of non-hydroxylic hydrogen atoms by metals might also occur in Fehling's and other similar solutions. In experiments with equal molecular quantities of dihydroxytartrates and tartrates, it had been found that the power of retaining copper in solution in the presence of alkali is very much greater in the latter case, although the reverse might be expected if replacement occurs in the alcoholic hydroxyl groups as is commonly supposed.

*71. "Properties and Relationships of Dihydroxytartaric Acid. Part II. Metallic Salts." By HENRY J. HORSTMAN FENTON, M.A.

The only metallic dihydroxytartrate which appears to have been prepared and studied is the sodium salt—the supposed barium salt being probably a mixture. Several other salts of the acid are now described. It is shown that the solubilities of the normal salts of sodium, potassium, rubidium, and caesium increase with the rise in atomic weight of the metal. The lithium salt is exceptional, its solubility being greater than that of the sodium salt. The differences in solubility of these salts may perhaps be of service in separating the metals of the alkalis.

Solutions of soluble dihydroxytartrates act as powerful reducing agents towards salts of silver, copper, and mercury, but are themselves reduced to dihydroxymaleic acid by stannous and ferrous salts.

72. "The Affinity-constants of Dihydroxymaleic, Dihydroxyfumaric, Dihydroxytartaric, and Tartaric Acids." By S. SKINNER, M.A.

The affinity-constants have been determined from the conductivity of the aqueous solutions. The determination in the case of these acids, with the exception of tartaric acid, presents special difficulty as their solutions undergo gradual decomposition. The constants found are compared with those of malonic, succinic, fumaric, maleic, and tartaric acids, and it is shown that an increase in the value of the constant occurs—(1) on the introduction of hydroxyl groups; (2) for the lower members of the series of dibasic acids; (3) for the unsaturated acids as compared with their saturated isologues.

DISCUSSION.

Dr. WALLACE WALKER objected to the explanation given of the nature of the curves, on the ground that in the production of carbon dioxide and glycollic aldehyde from dihydroxymaleic acid, the concentration of the supposed catalyst is decreasing at the same rate as that of the negative ion. The reaction would therefore be bimolecular, and its curve not rectilinear but one represented by an equation of the second degree.

Mr. FENTON asked whether the results obtained with dihydroxymaleic acid could be explained by the suggestion, advanced in a previous communication, that combination with a molecular proportion of water occurs on dissolution with the production of the unstable trihydroxy-succinic acid.

Mr. SKINNER, in reply, recognised that explanations of the behaviour of dihydroxymaleic acid other than that adopted in the paper were possible, but he was not prepared to say how far they would be of assistance in elucidating the results of the conductivity experiments.

73. "Notes on the Enolic and Ketonic Forms of Ethylic Acetoacetate." By R. S. MORRELL, M.A., Ph.D., and J. M. CROFTS, B.A., B.Sc.

The authors find that the purple oil obtained by the action of anhydrous ferric chloride on ethylic acetoacetate is decomposed by water in the presence of ether into ferric chloride and the ketonic ethereal salt. The ether is found to contain the ketonic and not the enolic form, as would be expected. To identify the ketonic form, the method given by Schiff and Bertini (*Ber.*, 1898, xxxi., 207) is employed. The ethylic benzalanilineacetoacetate is found to have the composition, melting-point (78°), and properties of the ketonic form. The tautomeric change is probably due to the fact that the ferric chloride compound of ethylic acetoacetate, when decomposed by water in the presence of ether, produces hydrochloric acid, which converts the enolic into the ketonic form.

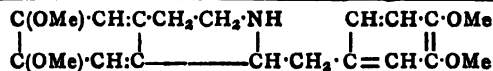
The authors mentioned in a previous paper that ethylic benzaldiacetoacetate formed a purple oil with ferric chloride in benzene solutions, whereas in alcoholic solution there was no such colouration. This oil has been obtained in the solid form, and is being investigated on the lines just indicated for the ethylic acetoacetate compound.

74. "The Resolution of Tetrahydropapaverine into its Optically Active Components." By WILLIAM JACKSON POPE and STANLEY JOHN PRACHEY.

Goldschmiedt has shown that papaverine is optically inactive, and is, in all probability, an isoquinoline derivative of the constitution—



This formula does not contain an asymmetric carbon atom, and, up to the present, no optically active derivative of papaverine has been described. Amongst the derivatives of papaverine prepared by Goldschmiedt (*Monats.*, 1886, vii., 493) is a tetrahydropapaverine, $\text{C}_{20}\text{H}_{25}\text{NO}_4$, to which, on the basis of the above constitution, the formula



must be assigned, since, on reducing isoquinoline derivatives, hydrogenation takes place in the pyridine nucleus. On this assumption, tetrahydropapaverine contains an asymmetric carbon atom, and it should therefore be possible to separate the optically inactive compound into two enantiomorphously related isomerides, and thus obtain evidence confirming the constitution which Goldschmiedt has assigned to papaverine.

In "splitting" an externally compensated base into its enantiomorphously related components, the dextro-tartrate is almost invariably employed; it would seem preferable, however, on various grounds, to make use in such cases of the dextrobromocamphorsulphonic acid described by Kipping and Pope (*Trans.*, 1893, lxiii., 584). Dextrobromocamphorsulphonic acid is a far more powerful acid than tartaric acid, and hence might be expected to yield better characterised and more easily isolated salts than the latter, with the further advantage that with a monobasic acid there is usually no tendency to form two classes of salts. Dextrobromocamphorsulphonic acid is conveniently prepared from the ammonium salt by conversion into the barium salt and careful precipitation of the solution by sulphuric acid. After filtration, the strength of the resulting solution is determined by titration.

Tetrahydropapaverine is boiled with rather more than the calculated quantity of dextrobromocamphorsulphonic acid dissolved in a large quantity of water. Ultimately the whole dissolves, and, on cooling, long needles of lævo-tetrahydropapaverine dextrobromocamphorsulphonate,— $\text{C}_{20}\text{H}_{25}\text{NO}_4 \cdot \text{C}_{10}\text{H}_{14}\text{BrO} \cdot \text{SO}_3\text{H}$,

crystallise out. Further quantities of this salt are obtained, mixed with increasing quantities of the more soluble dextrotetrahydropapaverine dextrobromocamphorsulphonate, on concentrating the mother-liquors. The two salts are readily separated by crystallisation from boiling water.

Lævo-tetrahydropapaverine dextrobromocamphorsulphonate crystallises readily from water, in which it is sparingly soluble in the cold, in long, colourless, anhydrous needles, melting with decomposition at 295–298°; it is somewhat more soluble in absolute alcohol, but insoluble in most other organic solvents. In a 0.3 per cent absolute alcoholic solution, it has the specific rotation of about $[\alpha]_D = -30^\circ$.

Dextrotetrahydropapaverine dextrobromocamphorsulphonate separates from solution as a resin which could not be obtained crystalline; when purified by repeated separation from its boiling aqueous solution and dried in the air, it forms a gritty, amorphous powder.

Lævo-tetrahydropapaverine, $\text{C}_{20}\text{H}_{25}\text{NO}_4$, separates on cooling a hot aqueous solution of its dextrobromocamphorsulphonate to which excess of ammonia has been added; it crystallises from boiling dilute alcohol in colourless, minute, six-sided plates containing water, which melt at 223°. The large flat face of the crystal is perpendicular to the acute bisectrix of a very small axial angle and of negative double refraction; the material has the specific rotation $[\alpha]_D = -149.5^\circ$ in a chloroform solution containing 0.7987 grm. per 25 c.c. The dextrotetrahydropapaverine separated from its dextrobromocamphorsulphonate resembles the lævo-isomeride in all respects, but is of opposite rotation.

Inactive tetrahydropapaverine melts at 200–201°, and at ordinary temperatures is crystallographically different from its optically active components; it is therefore a racemic compound in the sense of Kipping and Pope's definition (*Trans.*, 1897, lxxi., 989). It is formed on crystallising a mixture of equal weights of the two optically active isomerides from dilute alcohol.

The salts of racemic, dextro- and lævo-tetrahydropapaverine are being investigated, and experiments on other optically inactive alkaloids and their reduction products are in progress.

75. "Molecular Weights of Permanganates, Perchlorates, and Periodates in Solution." By J. MURRAY CROFTS, B.A., B.Sc.

The molecular weight of potassium permanganate in solution has been determined only by the specific conductivity method. In view of the close relationship between manganese and chlorine indicated by the periodic classification of the elements, it was thought that further investigation of the molecular weights of these substances in solution would be of interest.

The method adopted was that devised by Löwenherz (*Zeit. Physik. Chem.*, 1895, xviii., 70), in which Glauber's salt is the solvent employed. In this solvent sodium salts cannot undergo dissociation. The methods by which practically pure crystalline sodium permanganate and the very deliquescent sodium perchlorate were obtained are described. Determinations with both the sodium and the potassium salts of the acids were made, and the results point to the formula $M'MnO_4$ for permanganates, and $M'ClO_4$ for perchlorates. For periodates (metaperiodates) the potassium salt only was used, and the constant obtained indicated KIO_4 as the formula.

The results afford additional evidence for the inclusion of manganese with the halogens in Group VII. of the periodic classification. The author is inclined to advocate Spring's formula for perchloric acid (*Bull. Acad. Belg.* [ii.], xxxix., 887), and a corresponding expression for permanganic acid, the chlorine and manganese atoms being regarded as heptavalent in these compounds.

76. "The Action of Chlorine on Pyridine." By W. J. SELL, M.A., F.I.C., and F. W. DOOTSON, M.A.

In this communication the results are given of a repetition, in part, of the work of Keiser (*Am. Chem. Y.*, 1886, viii., 308), undertaken primarily with the object of comparing his dichloropyridine hydrochloride with the trichloropyridine, melting at $71-72^\circ$, obtained by the authors by the action of phosphorus pentachloride on pyridine (*Proc.*, 1898, xiv., 110).

In consequence of the results obtained, the work was somewhat extended, and it is shown that (1) an additive compound of chlorine and pyridine is formed, the constitution of which is not yet established; (2) Keiser's dichloropyridine hydrochloride is a trichloropyridine; (3) others of the chloropyridines seem to be formed in small amount; (4) the compound, C_5H_5NCl , described by Keiser as an addition product of chlorine and pyridine, is pyridine hydrochloride.

77. "The Oxidation of Paranitrotoluenesulphonic Acid to Dinitrostilbenedisulphonic Acid and to Paranitrobenzaldehydorthosulphonic Acid." By R. HERZ and W. H. BENTLEY.

In this paper the authors describe their experiments on the oxidation of sodium paranitrotoluenesulphonate by sodium hypochlorite. By using much less caustic soda than Green and Wahl (*Ber.*, 1897, xxx., 3097), they find that the product of oxidation is almost entirely sodium dinitrostilbenedisulphonate, and that sodium dinitrobenzylsulphonate does not appear to be produced. The sodium, silver, barium, and lead salts, as well as the acid, have been prepared and examined.

The authors are unable to accept the views of Ris and Simon (*Ber.*, 1898, xxxi., 354) on the constitution of the stilbene derivative, and, from a determination of the amount of potassium permanganate required for its oxidation, maintain that it is a dinitro- and not a nitroso-nitro-compound. They found, independently of Green and Wahl, that, on oxidising sodium dinitrostilbenedisulphonate in the cold with potassium permanganate, sodium paranitrobenzaldehydorthosulphonate is formed. This substance possesses the properties of an aldehyde, and forms yellow crystalline compounds with phenylhydrazine and semicarbazide.

78. "Determination of Molecular Weights: Modification of Landsberger's Boiling-point Method." By JAMES WALKER and JOHN S. LUMSDEN.

The authors propose a modification of Landsberger's method (*Ber.*, 1898, xxxi., 458), which consists essentially in measuring the volume of the solution after the boiling-point has been determined, instead of ascertaining its weight. This is effected by graduating the boiling tube, and permits of a number of successive experiments being made with the same quantity of substance. Only one weighing is necessary for the whole series, which occasions a considerable saving of time, making it possible to obtain four or five determinations in the course of half an hour. The accuracy is comparable with that of the Viöor Meyer vapour density method in ordinary circumstances.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société Chimique de Paris.
Series 3, Vol. xix.-xx., No. 4.

On certain Molecular Changes between Soluble Salts capable of giving other Soluble Salts in a State of Purity. Conditions necessary; Applications.—E. Roca.—Not suitable for abstraction.

A Reaction of Aldehyds and Acetones.—MM. Lumière and Seyewitz.—If into a solution of 1000 grms. of water, 50 grms. of anhydrous sulphite of soda, and 10 grms. of pyrogallol acid we plunge a bromo-gelatin photographic plate which has had a normal exposure, it will be a long time, varying with the alkalinity of the sulphite and the sensibility of the plate, before an image appears; if, however, to the above solution we add successively various solutions of aldehyds or acetones, they will all considerably decrease the time which elapses before the image appears. The experiments were in every case made with 50 c.c. of the pyrogallol solution, and to this was added 1 grm. of either aldehyd or acetone dissolved in 25 c.c. of water or in 25 c.c. of alcohol when the compound was insoluble in water. A list of various aldehyds and acetones which have been tried is then given. The process can also be used for testing for certain compounds, and by its means, with the proper precautions, 1/25,000th part of methanal, 1/15,000th part of ethanal, and 1/2500th part of propanone have been detected.

Application of the Method of Friedel and Crafts for the Preparation of Aromatic Acetenes and Aldehyds in vacuo. Reply to M. Bouveault.—A. Verley.—A controversial paper, not suitable for abstraction.

Action of Chloride of Acetyl on Acetate of Phenyl in the presence of Chloride of Aluminium. Preparation of Para-acetate of Phenyl.—A. Verley.—To about 3 molecules of acetate of phenyl, 1 molecule of chloride of aluminium is introduced, taking care to prevent all rise of temperature. The apparatus is exhausted, and then, drop by drop, one molecule of chloride of acetyl is added; the temperature should not exceed 20° . When no more HCl gas comes off, the contents are precipitated by water, and a substance boiling at 160° at 23 m.m. is distilled off. When crystallised from alcohol it appears under the form of brilliant white crystals and melts at 54° . By oxidation with dilute boiling nitric acid it is transformed into acetylparaoxybenzoic acid melting at 185° .

The Migration of the Atom of Iodine in the Nitration of Aromatic Derivatives. (III.).—F. Reverdin (in collaboration with K. Racer).—A long paper, not suitable for abstraction.

Action of Ethylic Aldehyd on Phenylhydrazin.—H. Causse.—The experiments here described show that ethanal acting on phenylhydrazin gives birth to two products. One is hydrazone, fusible at 99° , and is the first

formed; the second is triethylidenediphenylhydrazine fusible at 109–110°.

Estimation of Oxygen Dissolved in Water.—MM. Albert Levy and F. Marboutin.—A reply to a criticism of a previous paper of theirs by M. Sokoloff in a Russian journal, the *Bparr*.

On the Essence of Basilic.—J. Dupont and M. Guerlain.—Under this name are included two products with very different properties, according to the place of their origin. That from France and Germany possesses a sweet characteristic odour; while that which comes from Reunion has, in addition, a strong odour of camphor, which nearly hides the other. Further, the first is levogyre and the second dextrogyre. After a number of other experiments, the authors are satisfied that chemically the essences from the plants cultivated in Europe are identical, while that from Reunion is from a different plant.

Journal de Pharmacie et de Chimie,
Series 6, vol. vii., No. 3.

The Estimation of the Phosphoglycerates.—MM. Adrian and Trillat.—The authors have verified the fact that glycerin does not interfere with the titration by means of indicators. For the estimation of phosphoric acid in the presence of glycerin, or of a neutral or acid phosphoglycerate or *vice versa*, an acid solution of phosphoglycerate of lime was prepared of such strength that 10 c.c. of the solution required 0.5 c.c. of potash to titrate it with heliantine, or 2.2 c.c. with phthalein. A mixture was made containing 10 c.c. of this solution, 10 c.c. of titrated phosphoric acid, and 10 c.c. of a solution of acid glycerophosphate and a few drops of glycerin. When titrated, this mixture required—With heliantine, 7.3 c.c.; with phthalein, 16.1 c.c. According to these figures, the sum of the acidities is divided in the following manner:—

	Heliantine.	Phthalein.
Acid glycerophosphate	0.5 c.c.	2.2 c.c.
Phosphoric acid	6.9 "	13.9 "

Total 7.4 " 16.1 "

Study of the Reaction of Phosphoric Acid on Glycerin.—MM. Adrian and Trillat.—The authors have endeavoured to prepare phosphoglyceric acid by Pelouze's method, and they obtained a product which on analysis partly corresponded to the acid phosphoglycerate of baryta. They also tried to decompose phosphoglycerate of potash by tartaric acid, as well as by other methods, but the result of their observation tends to show that there is not yet any known method of preparing phosphoglyceric acid in a state of purity; that this acid is decomposed by heat, and even by simple concentration, forming phosphoric acid; that the reaction of phosphoric acid on glycerin is complex, though limited; that there exists in conjunction with phosphoglyceric acid at least one diether, neutral to indicators, formed of one molecule of acid for two of glycerin, very slowly decomposable in the cold by the alkaline carbonates.

MEETINGS FOR THE WEEK.

- MONDAY, 23rd.—Society of Arts, 8. (Cantor Lectures). "Electric Traction," by Prof. Carus Wilson.
- TUESDAY, 24th.—Royal Institution, 8. "The Historical Development of Modern Europe," by Samuel Rawson Gardiner, M.A., D.C.L., LL.D.
- Society of Arts, 8. "The Goldfields of British Columbia," by W. Hamilton Merritt.
- THURSDAY, 26th.—Royal Institution, 8. "Heat," by The Right Hon. Lord Rayleigh, F.R.S., &c.
- FRIDAY, 27th.—Royal Institution, 9. "Sir Stamford Raffles and the Malay States," by Lieut.-Gen. the Hon. Sir Andrew Clarke, R.E.
- Physical, 5. "A simple Interference Method of Reducing Prismatic Spectra," by Messrs. Edser and Butler. "Some further Experiments on the Circulation of the Residual Gaseous Matter in Crookes Tubes," by Mr. Campbell Swinton.
- SATURDAY, 28th.—Royal Institution, 3. "The Biology of Spring," by J. Arthur Thomson, M.A.

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CONTENTS.

ARTICLES:—	PAGE
Fifth Annual Report of Committee on Atomic Weights—Results Published during 1897, by F. W. Clarke.....	239
Gladding's Method for Phosphoric Acid, by John B. Coppock, F.C.S.....	242
The Preparation of Carbonate of Menthol, by M. Erdmann	242
On the Estimation of Manganese Separated as the Carbonate, by Martha Austin	242
The Dissociation of Electrolytes as Measured by the Boiling- point Method, by Harry C. Jones and Stephen H. King	243
On the Separation of the Oxides of Cerium and Thorium, by G. Wybournoff and A. Vernouil	245
PROCEEDINGS OF SOCIETIES—	
PHYSICAL SOCIETY.....	246
NOTICES OF BOOKS.—Text-book of Physical Chemistry—Notes on Observations—The Proceedings of the Chemical and Metallurgical Society of South Africa—The Elements of Electro-chemistry treated Experimentally	247
CHEMICAL NOTICES FROM FOREIGN SOURCES	248
MISCELLANEOUS	250
MEETINGS FOR THE WEEK	250

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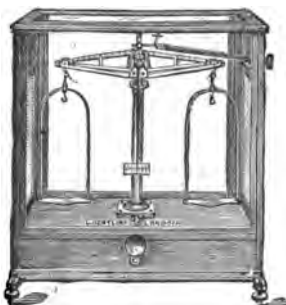
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THE CHEMICAL NEWS.

VOL. LXXVII., No. 2009.

FIFTH ANNUAL REPORT OF COMMITTEE ON ATOMIC WEIGHTS. RESULTS PUBLISHED DURING 1897.*

By F. W. CLARKE.

DURING 1897 comparatively few new determinations of atomic weights have appeared. The data given are as follows:—

Carbon.

Scott (*Journ. Chem. Soc.*, lxxi., 550, May, 1897) has carefully studied the determinations of the atomic weight of carbon which depend upon the combustion of the element and the weight of the dioxide formed, and has discovered an important correction. This is due to the considerable change of volume in the potash solution produced by the absorption of carbon dioxide, which involves a notable change in the reduction of the weighings to a vacuum. Scott has measured the amount of this change, and has applied it to the work of his predecessors with the subjoined results. In each case the values given are the means of the several experiments recorded.

	Uncorrected.	Corrected.
Dumas and Stas	11'9975	11'9938
Erdmann and Marchand.	12'0093	12'0054
Roscoe	12'0029	11'9973
Friedel	12'0112	12'0056
Van der Plaats.	12'0031	12'0018
Mean.. ..	12'0048	12'0008

These values are based upon O=16; with O=15'879, the corrected mean result becomes C=11'910.

Scott further criticises in detail the determinations by Stas which depend upon the combustion of carbon monoxide, and shows that they require possible corrections and involve various uncertainties. He concludes "that the atomic weight of carbon is by no means one of those most accurately known, and that a careful revision of it is imperative."

Nitrogen, Chlorine, and Silver.

Leduc (*Comptes Rendus*, cxv., 299, August 2, 1897), on the basis of his density determinations, assumes N=14'005 when O=16. Applying this to the determinations by Stas of the ratios Ag:Cl, AgCl:O₃, and AgCl:NH₄Cl, he finds a discrepancy of about 1 part in 320. That is, the work of Stas gives N=14'044 instead of 14'005. This disagreement Leduc attributes to the presence of occluded oxygen, as pointed out by Dumas, in the silver used by Stas. For this he assumes a correction, and then, combining his value for nitrogen with the data given by Stas, he computes H=1'0076, Cl=35'470, and Ag=107'916. The correction applied by Leduc seems to be somewhat uncertain; and it is doubtful whether his determination of the density of nitrogen is entitled to greater weight than the other data which are involved in the calculations.

Aluminum.

In the third and fourth reports of this committee, Thomsen's data for the atomic weight of oxygen are given. In these determinations oxygen and hydrogen were compared with aluminum as an intermediary; but the aluminum used was not absolutely pure. Had it been

pure, the atomic weight of the metal, with reference to the two gases, could have been computed.

Thomsen now (*Zeit. Anorg. Chem.*, xv., 447) gives the data necessary for the comparison, and deduces the atomic weight of aluminum. The impurities are determined and corrections for them applied. There is also a correction for the change in volume of the potash solution in which the metal was dissolved. Applying the corrections, Thomsen finds that 0'99897 grm. pure aluminum correspond to 0'11195 grm. of hydrogen. Hence, with H=1,—

$$Al = 26'770.$$

Again, 0'99897 grm. of metal is equivalent to 0'88824 grm. of oxygen. Hence, with O=16,—

$$Al = 26'992.$$

These two values, referring to the two standard units, are determined independently of each other, and are independent of any measured ratio between O and H. They are notably lower than the values computed by myself from Mallet's data, 26'91 and 27'11, and possibly should supplant the latter. This question, however, needs further discussion, and probably some additional experiments.

Nickel.

Atomic weight determined by Richards and Cushman (*Proc. Amer. Acad.*, xxxiii., 97, November, 1897), from analyses of the anhydrous sublimed bromide, NiBr₂. This substance is shown by the authors to be perfectly suited to the purpose of the investigation. The method of analysis was in all essential respects that used by Richards in other researches of the same kind, and was partly gravimetric and partly volumetric. On the one hand, the ratio 2AgBr:NiBr₂ was determined; on the other, the ratio 2Ag:NiBr₂. All weighings were reduced to a vacuum. The results, in three series, were as follows:—

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2'80668	4'82431	58'708
1'41317	2'42880	58'716
1'71759	2'95307	58'650
2'48565	4'27357	58'651
4'32997	7'44280	58'700
2'18072	3'74856	58'693

Mean.. .. 58'680

SECOND SERIES.

Weight NiBr ₂ .	Weight AgBr.	Atomic weight Ni.
3'28039	5'63892	58'691
2'70044	4'64208	58'686
3'38230	5'81391	58'698
1'33459	2'29435	58'670
1'25054	2'14963	58'693
1'32778	2'27384	58'690
2'24452	3'85805	58'705

Mean.. .. 58'690

THIRD SERIES.

Weight NiBr ₂ .	Weight Ag.	Atomic weight Ni.
3'28039	3'23910	58'701
2'70044	2'66636	58'709
3'38230	2'33990	58'689
1'33459	1'31787	58'689
1'25054	1'23482	58'698
1'32278	1'30629	58'675
2'24452	2'21652	58'676

Mean.. .. 58'691

From all these data the authors conclude that the atomic weight of nickel cannot be far from 58'69, when O=16, or 58'25 if O=15'88.

* From the *Journal of the American Chemical Society*, xx., No. 3.

From series two and three 15.32086 grms. of silver give 26.67078 grms. of bromide. Hence the percentage of silver in silver bromide is 57.444, which agrees with the value 57.445 found by Stas. This is a good check upon the accuracy of the work.

Cobalt.

The atomic weight determinations by Richards and Baxter (*Proc. Amer. Acad.*, xxxiii., 115, December, 1897) are in all essential respects like those of nickel which have just been cited. The three series of data are as follows:—

FIRST SERIES.		
Weight CoBr ₂	Weight AgBr.	Atomic weight Co.
2.25295	3.86818	58.950
2.88763	4.95732	58.957
1.88806	3.24056	59.026

Mean.. .. 58.984

SECOND SERIES.		
Weight CoBr ₂	Weight AgBr.	Atomic weight Co.
1.33564	2.29296	58.975
2.58129	4.43095	58.998
2.84382	4.88135	59.009
1.83722	3.15368	59.000
2.68584	4.61046	58.996
3.18990	5.47607	58.982
2.88914	4.95943	58.997
2.32840	3.99706	58.987
1.91703	3.29053	59.010

Mean.. .. 58.995

THIRD SERIES.		
Weight CoBr ₂	Weight Ag.	Atomic weight Co.
1.33564	1.31702	59.002
2.58129	2.54585	58.955
2.84382	2.80449	58.977
1.83722	1.81170	58.991
2.68584	2.64879	58.969
2.88914	2.84891	58.998
2.32840	2.29593	59.003
1.91703	1.89033	58.999

Mean.. .. 58.987

If O=16, Co=58.99, as the outcome of all the series. If O=15.88, Co=58.55. From the data in series two and three the percentage of silver in silver bromide is 57.446, while Stas gives 57.445. The last five experiments, which are the best, give 57.447. This check on the accuracy of the work is therefore satisfactory.

Tungsten.

The investigation by Hardin (*Journ. Am. Chem. Soc.*, xix., 657, August, 1897) is rather the scrutiny of a method than an attempt to accurately fix an atomic weight. Most of the determinations of the atomic weight of tungsten have been based upon analyses or syntheses of the trioxide; and this substance is now shown to be exceedingly untrustworthy as regards the measurement of this constant. Hardin has made sixty-four determinations with the trioxide, in seventeen groups or series, using material from various sources, and working under a great variety of conditions. The values found range from W=183.51 to W=185.00 when O=16; the average of all being 184.106. The same discordance appears upon comparing the results obtained by previous investigators, and it seems to be partly due to absorptions of tungsten or its oxide by the boat in which the reductions or oxidations were effected, and partly to the presence of nitrogen in the trioxide when the latter was prepared from ammonium tungstate. At all events, the method is unsuited to its purpose, and the atomic weight of tungsten needs thorough re-investigation by means of other processes.

Cerium.

The atomic weight of this metal has been re-determined by Wyrnonboff and Verneuil (*Bull. Soc. Chim.*, [3], xvii., 679) upon material purified by an elaborate process and with extreme care. From this material the oxide CeO₂ was obtained perfectly white—a result in corroboration of the researches of Wolf and of Wing, but contrary to later investigators who have always found the substance to be more or less coloured. The existence of a white oxide is also confirmed by the recent investigations of Moissan (*Comptes Rendus*, cxiv., 1233).

The salt chosen for the atomic weight determinations was the sulphate, Ce₂(SO₄)₃.8Aq, a compound which is perfectly definite and stable, and which is easily obtained in a state of purity. At 250° it loses its water with facility, and up to 500° it undergoes no further decomposition. By ignition at a white heat, 1500° approximately, all sulphuric acid is expelled, and the pure oxide remains. The other hydrated sulphates of cerium are less manageable. The material studied was derived from three distinct sources, partly from monazite, partly from cerite; and the results obtained confirm one another. The weights were as follows, but nothing is said as to their reduction to a vacuum. Probably the reduction was not made:—

FIRST SAMPLE.		
Hydrous salt.	Anhydrous salt.	CeO ₂ .
1.2385	0.9875	0.5977
1.2730	1.0148	0.6138
1.2030	0.9590	0.5794
1.5420	1.2295	0.7430

SECOND SAMPLE.		
0.9642	0.7685	0.4542
1.3260	1.0571	0.6389
1.1429	0.9112	0.5518
0.9072	0.7232	0.4372

THIRD SAMPLE.		
1.2114	0.9658	0.5840
1.2411	0.9894	0.5984

From these data the following atomic weights are derived when O=16 and S=32, cerium being regarded by the authors as a dyad, with the oxide obtained having the formula Ce₂O₄.

FIRST SERIES.		
From per cent H ₂ O.	From "Ce ₂ O ₄ " in hydrate.	From Ce ₂ O ₄ in anhydrous salt.
92.84	93.08	93.16
92.65	92.88	92.95
92.65	92.64	92.63
92.85	92.74	92.70
Mean ..	92.74	92.86

SECOND SERIES.		
92.49	92.55	92.56
92.69	92.72	92.73
92.76	93.17	93.30
92.66	92.77	92.80
Mean ..	92.65	92.85

THIRD SERIES.		
92.75	92.84	92.87
92.68	92.88	92.93
Mean ..	92.71	92.90
Mean of all	92.70	92.87

The extreme variation is 0.75, which becomes 1.125 when cerium is regarded as a triad.

From the entire series of determinations, the salt $\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{Aq}$ gives in mean 20.278 per cent of water, and 48.205 CeO_2 . The mean percentage of CeO_2 in the anhydrous sulphate is 60.467. From these data, with $\text{H}=1$, $\text{O}=15.879$, and $\text{S}=31.83$, the following mean values for Ce^{III} are deduced:—

From percentage of water $\text{Ce}=138.14$
From CeO_2 in hydrated salt $\text{Ce}=138.35$
From CeO_2 in $\text{Ce}_2(\text{SO}_4)_3$ $\text{Ce}=138.42$

The mean of these is $\text{Ce}=138.30$. With $\text{O}=16$, this becomes $\text{Ce}=139.35$.

These figures are adopted in the table at the close of this report, as being probably more trustworthy than the older determinations, but they are still subject to a good deal of uncertainty.

Still another investigation upon cerium is reported by Boudouard (*Comptes Rendus*, cxv., 772), who, following Schützenberger, fractionates the salts of the metal. His atomic weight determinations are made by ignition of the sulphate; but the values assigned to O and S are not stated. By fractionation of the acetate he gets an oxide giving from $\text{Ce}=135.1$ to 140.7 . From fractionations of the sulphate he finds $\text{Ce}=133.0$ to 138.75 . Hydrogen peroxide precipitates an oxide giving $\text{Ce}=137.15$ to 137.6 , while the unprecipitated portion ranges from 137.85 to 139.9 . He concludes that the oxide of cerium is accompanied by small quantities of another earth of lower atomic weight.

Wyruboff and Verneuil (*Comptes Rendus*, cxv., 950) criticise the work of Boudouard, and suggest that his high values for Ce are due to the presence of thorium, while the lower values are ascribable to the other earths of the cerium-yttrium groups. To this criticism Boudouard replies (*Comptes Rendus*, cxv., 1006), defending the purity of his material. He now states that his CeO_2 was white. Wyruboff and Verneuil (*Comptes Rendus*, cxv., 1180) reiterate their objections, and show that the variations in successive portions obtained by Boudouard are irregular, sometimes high, sometimes low, and not systematic in one direction as they should be with material representing an orderly fractionation.

It is evident from the discussion thus summarised that Boudouard's figures relate to mixtures, and not to one earth, whatever the mixtures may be. They are therefore unavailable as determinations of a definite atomic weight. It may be suggested here that a careful scrutiny of the supposed CeO_2 is necessary in order to prove that it is not contaminated by higher or lower oxides of cerium, and that it does not contain, like certain other oxides, occluded gaseous impurities. Until these questions have been settled, all atomic determinations based upon weighings of ceric oxide are uncertain.

Gaseous Densities.

Lord Rayleigh (*Nature*, December 30, 1897; *CHEMICAL NEWS*, December 31) has continued his investigation upon the density of the principal gases, and now gives data for CO , CO_2 , and N_2O . His results, air being taken as unity, are tabulated as follows:—

Oxygen 1.10535
Atmospheric nitrogen, plus argon. 0.97209
Nitrogen 0.96737
Argon 1.37752
Carbonic oxide 0.96716
Carbon dioxide 1.52909
Nitrous oxide 1.52951

Calculated from the density of Co , with $\text{O}=16$, $\text{C}=11.9989$.

Mathematical Relations.

Rummel (*Proc. Roy. Soc. of Victoria* (Australia), x., Part 1, p. 75), in a paper upon "The Spectra of the Alkalis and their Atomic Weights," works out a series of

	$\text{H}=1$.	$\text{O}=16$.
Aluminum.. ..	26.91	27.11
Antimony	119.52	120.43
Argon	?	?
Arsenic	74.44	75.01
Barium	136.39	137.43
Bismuth	206.54	208.11
Boron	10.86	10.95
Bromine	79.34	79.95
Cadmium	111.10	111.95
Cæsium	131.89	132.89
Calcium	39.76	40.07
Carbon	11.91	12.00
Cerium	138.30	139.35
Chlorine	35.18	35.45
Chromium	51.74	52.14
Cobalt	58.55	58.99
Columbium	93.02	93.73
Copper	63.12	63.60
Erbium	165.06	166.32
Fluorine	18.91	19.06
Gadolinium	155.57	156.76
Gallium	69.38	69.91
Germanium	71.93	72.48
Glucinum	9.01	9.08
Gold	195.74	197.23
Helium	?	?
Hydrogen	1.000	1.008
Indium	112.99	113.85
Iodine	126.89	126.85
Iridium	191.66	193.12
Iron	55.60	56.02
Lanthanum	137.59	138.64
Lead	205.36	206.92
Lithium	6.97	7.03
Magnesium	24.10	24.28
Manganese	54.57	54.99
Mercury	198.49	200.00
Molybdenum	95.26	95.99
Neodymium	139.70	140.80
Nickel	58.24	58.69
Nitrogen	13.93	14.04
Osmium	189.55	190.99
Oxygen	15.88	16.00
Palladium	105.56	106.36
Phosphorus	30.79	31.02
Platinum	193.41	194.89
Potassium	38.82	39.11
Praseodymium	142.50	143.60
Rhodium	102.23	103.01
Rubidium	84.78	85.43
Ruthenium	100.91	101.68
Samarium	149.13	150.26
Scandium	43.78	44.12
Selenium	78.42	79.02
Silicon	28.18	28.40
Silver	107.11	107.92
Sodium	22.88	23.05
Strontium	86.95	87.61
Sulphur	31.83	32.07
Tantalum	181.45	182.84
Tellurium	126.52	127.49
Terbium	158.80	160.00
Thallium	202.61	204.15
Thorium	230.87	232.63
Thulium	169.40	170.70
Tin	118.15	119.05
Titanium	47.79	48.15
Tungsten	183.43	184.83
Uranium	237.77	239.59
Vanadium	50.99	51.38
Ytterbium	171.88	173.19
Yttrium	88.35	89.02
Zinc	64.91	65.41
Zirconium	89.72	90.40

relations which seem to be significant. For the formulae developed, which are algebraic, the original paper must be consulted. When $\text{Na}=23$, as the starting-point, the spectral relations give $\text{He}=3.64$, $\text{K}=39.05$, $\text{Rb}=85.15$, and $\text{Cs}=133.47$. These values are near enough to the actual determinations to show that the mathematical process deserves careful consideration.

Miscellaneous Notes.

At the Toronto Meeting of the British Association for the Advancement of Science, Brauner presented a paper on the atomic weight of thorium. Also at the Washington Meeting of the American Chemical Society, Venable gave the preliminary results of an investigation upon zirconium (see *CHEMICAL NEWS*, vol. lxxvii., p. 221).

A memoir upon the atomic weight of molybdenum by A. Vandenberghe, has been awarded the Stas prize of the Belgian Academy. It will appear in the quarto form adopted by the Academy for its more important publications.

The accompanying table of atomic weights is essentially that given in Clarke's "Re-calculation," a new edition of which was published by the Smithsonian Institution in January. The names of elements which represent changes from the table of 1896 are italicised.

GLADDING'S METHOD FOR PHOSPHORIC ACID.

By JOHN B. COPPOCK, F.C.S.,
Lecturer, Lancashire County Council Agricultural School.

In the *CHEMICAL NEWS* (vol. lxxvii., p. 32) Gladding gives a method for the gravimetric estimation of phosphoric acid as ammonium phosphomolybdate, which can be applied to fertilisers.

Although generally attempts to estimate the amount of phosphoric acid by precipitation and weighing as the yellow ammonium phospho-molybdate have not given reliable results, there seems to be no reason why Gladding's method should not be widely applied for phosphoric acid determination in fertilisers.

In some analyses which have recently passed through my hands, I have made comparisons between the magnesia method and the Gladding method. The point in favour of the Gladding method is the quickness of the determination.

The following results have been obtained:—

	Boiled Bones.	
	Magnesia method.	Gladding's method.
Sample I. . . .	25.22	25.80
" II. . . .	23.18	23.85

In each case the figures represent the percentage of phosphoric acid present. Correcting the figures in the magnesia method by adding 0.33 (the usual solubility allowance) we get—

25.55 per cent phosphoric acid
23.51 " " "

which shows that the new method is quite satisfactory for fertiliser work.

Superphosphates.

Superphosphates gave the following figures in two cases:—

	Magnesia method.	Gladding's method.
Sample I. . . .	16.34	16.50
" II. . . .	15.17	15.32

Thus, although the figures are invariably higher for Gladding's method, they are exact enough for fertiliser work. If the method is used the nitric acid can be added at once to the extent of 50 c.c. if the acid is of specific gravity 1.42 and the ammonia of specific gravity 0.88. The

solution is then quite acid enough for the precipitation of the yellow salt by the addition of 40 c.c. of the usual 5 per cent molybdic oxide solution. This is quite sufficient acid solution to ensure complete precipitation if 0.25 gm. of the fertiliser has been used.

My precipitates were dried first in the water-bath, then finished in the air-bath, despite the fact that the author of the method discarded his air-bath for a glycerol one.

Washing the precipitate off smoothened filter-paper into a platinum dish was tried, as in the determination of phosphoric acid in basic slag as ammonium phosphomolybdate. The results are quite satisfactory. There can be no doubt that the method affords a quick and reliable way for the determination of phosphoric acid in fertilisers.

THE PREPARATION OF CARBONATE OF MENTHOL.

By M. ERDMANN.

MENTHOL appears to have been used medicinally with some success during the past few years; but its use is often limited by the caustic action it has on the mucous membrane, so that some writers have proposed substituting carbonate of menthol instead.

This product has been described by G. Arth (*Ann. de Chim. et de Phys.*, 1886, vii., 469), who obtained it by the action of cyanogen gas or of chloride of cyanogen on sodium-menthol. The principal product of this reaction is menthylurethane, and carbonate of menthol only occurs as a by-product in small quantities.

The pyridine method, used to effect substitutions to the functional hydrogen of the terpenic alcohols, gives a rapid and convenient method of preparing carbonate of menthol. The method of operating is as follows. To a solution of—

Menthol	30 grms.
Chloroform	30 c.c.
Pyridine (anhydrous)	25 "

we add, by small portions, a chloroformic solution of phosgene containing 10 grms. of COCl_2 , stirring the whole time. This is then left to stand for twenty-four hours, then, without separating the two layers of liquid, it is treated by steam. Traces of uncombined menthol pass off at the same time as the pyridine. The residue consists of crystalline carbonate of menthol, which is washed with warm water and re-crystallised in a large quantity of warm alcohol. The carbonate, hardly soluble in alcohol in the cold, is deposited in long white prisms melting at 105° precisely. The reaction is almost quantitative.—*Journ. für Prakt. Chemie*, lvi., p. 43.

ON THE ESTIMATION OF MANGANESE SEPARATED AS THE CARBONATE.*

By MARTHA AUSTIN.

THE estimation of manganese precipitated as the manganous carbonate, when that salt is obtained by the action of sodium or potassium carbonate, has been regarded as very undesirable for the reasons that, even if the conditions of the precipitate is such that it does not run through the filter, the manganous carbonate can never be freed entirely from alkaline salt, and that the conversion of the carbonate to the manganosomanganic oxide—the form in which it is customary to weigh—is too uncertain. It had been supposed also that the presence of ammoniacal salts (as well as of carbonic acid) causes solution of the

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, vol. v., p. 388.

TABLE I.

MnCl ₂ . C.m.s.	MH ₂ Cl. Grms.	MnCO ₃ .			Mn ₂ O ₃ .		
		Found.	Theory.	Error.	Found.	Theory.	Error.
50	10	0.2685	0.2680	0.0005 +	0.1770	0.1776	0.0006 -
50	10	0.2704		0.0024 +	0.1788		0.0012 +
50	10	0.2710		0.0030 +	0.1770		0.0006 -
50	10	0.2720		0.0040 +	0.1774		0.0002 -

TABLE II.

	MH ₂ Cl. Grm.	Mn ₂ O ₃ .			MnSO ₄ .		
		Found.	Theory.	Error.	Found.	Theory.	Error.
1.	10	0.2463	0.2478	0.0015 -	0.4903	0.4905	0.0002 -
2.	10	0.1110	0.1121	0.0011 -	0.2225	0.2219	0.0006 +
3.	10	0.1584	0.1581	0.0003 +	0.3126	0.3128	0.0002 -
4.	10	0.1672	0.1699	0.0027 -	0.3355	0.3364	0.0009 -

manganous carbonate, until the work of Guyard (Hugo Tamm) (CHEMICAL NEWS, xxvi., 37) showed that when the precipitation is accomplished by ammonium carbonate, even in the presence of ammonium chloride, complete separation of the manganese is possible. No data are given by Guyard to show the completeness of the separation of the manganese by this process; but Fresenius (*Zeit. f. Anal. Chemis.*, 1872, 290) examined the method and speaks favourably of it. In this process the main difficulty of the older method of estimation of the carbonate—viz., the inclusion of the alkaline salt—is avoided. We know now how to avoid the difficulty in the way of weighing as the oxide by converting that substance to the form of the sulphate, as shown in a former paper (*Am. Journ. Sci.*, IV., v., 209).

For a careful study of the separation of manganous carbonate by Guyard's method a solution of pure manganous chloride was prepared and standardised as the anhydrous sulphate in the manner detailed in the paper to which reference has been made above. A definite volume of the manganous chloride was carefully drawn into a platinum dish and diluted to a volume of 200 c.c. To the solution heated to 100° C. ammonium chloride (about 10 grms.) was added and ammonium carbonate in excess. The solution was kept warm until the precipitate subsided, and then was filtered off on asbestos on a perforated crucible under pressure. The presence of ammonium chloride is necessary to insure such a condition of the precipitate that it will not run through the felt.

Inasmuch as the precipitate was collected under conditions which readily permit an attempt to weigh as the carbonate, a trial of that method was made incidentally. The event proved as Rose (*Ann. Phys. Chem.*, 1851, lxxxiv., 52) has stated previously, that when the carbonate is gently heated, evolution of carbon dioxide and oxidation of the residue begins before the water is thoroughly removed; for, though nearly all the results are above the theory, the solution of the residue in hydrochloric acid indicated plainly the presence of a small amount of a higher oxide of manganese. In Table I. are found the results of a series of experiments in which the attempt was made to weigh first as carbonate and again after strong ignition—well within the oxidising flame of a powerful burner (*Am. Journ. Sci.*, IV., v., 209)—as the manganoso-manganic oxide. The application of the bromine test to the hot ammoniacal filtrate showed that, in every one of these experiments, the precipitation of the manganese in the form of the carbonate had been complete.

As shown in Table I., weighing as the carbonate is out of the question; the errors of the process when the residue is ignited in the manner described to form the manganoso-manganic oxide are much smaller, though rather variable. The estimation of manganese as the anhydrous sulphate had given, in the work to which reference has been made, results agreeing so much more closely than could be obtained by any other method of procedure, that the attempt was made to estimate the amount of manganese precipi-

tated as the manganese carbonate by converting it first to the oxide, then to the sulphate. A given weight of sulphate was precipitated as the manganous carbonate, after the employment of all the precautions mentioned previously in this paper, and then filtered off on ashless filter-paper. After washing thoroughly with hot water, the filter was burned, the residue ignited for the condition of the manganoso-manganic oxide and weighed as such. Then the oxide was converted to the sulphate by heating with three or four drops of concentrated sulphuric acid. The agreement of the results, as shown in Table II., is considerably better.

By treatment of the filtrates of 1, 2, and 3 with bromine and ammonia at boiling temperature, no manganese was found. In the filtrate from No. 4, by the same treatment, a small amount of manganese dioxide was precipitated, which, when heated with concentrated sulphuric acid, gave 0.0006 gm. of manganous sulphate; and, hence, the error in that determination is really 0.0003 gm. on the sulphate. The slightly larger deficiency recorded in the table was probably due to imperfect filtering.

It seems to be evident that Guyard's method of separation of manganese as the manganous carbonate, when handled with precautions, gives complete separation of that element. It must be recognised clearly, however, that the precipitation should be made in the presence of a considerable amount of ammonium chloride, and that great care must be used in the filtering and washing of the finely divided precipitate. It is altogether preferable to weigh in the condition of the sulphate.

In conclusion thanks are hereby expressed to Professor F. A. Gooch for kind assistance.

THE DISSOCIATION OF ELECTROLYTES AS MEASURED BY THE BOILING-POINT METHOD.*

By HARRY C. JONES and STEPHEN H. KING.

THE two methods of general application for measuring the amount of electrolytic dissociation in aqueous solutions, are, the conductivity method of Kohlrausch (*Ann. der Phys., Wiss.*, xxvi., 161), and the freezing-point method of Beckmann in its most improved forms (*Ibid.*, li., 500; *Zeit. Phys. Chem.*, xi., 529). There are, in addition, several methods available for determining the concentration of particular kinds of ions, or which admit of application to a limited number of cases, as the well-known solubility method of Nernst and Noyes (*Zeit. Phys. Chem.*, iv., 372; vi., 241). The two methods, which can be used satisfactorily with water solutions, are, in turn, of limited applicability when other solvents are employed. The use of conductivity as a direct measure of dissociation is most satisfactory when water is used

* From the *American Chemical Journal*, vol. xix., No. 9.

as a solvent. A difficulty which is encountered with other solvents is, that the dissociation in them is not complete at dilutions which come within the range of that method. The value of M_{∞} cannot, therefore, be determined directly, and some assumption, which may require proof, is sometimes made in an attempt to determine its approximate value. Considerable work has, however, already been done on the conductivity of solutions of electrolytes of different concentrations in solvents other than water, —notably the work of Fitzpatrick (*Phil. Mag.*, 377, 1887), Paschkow ("Über die Leitfähigkeit einiger Salze in Alkohol Lösungen," Charkow, 1892), and Völlmer (*Ann. der Phys., Wiss.*, lii., 328), on solutions in ethyl and methyl alcohol; of Zelinsky and Krapivin (*Zeit. Phys. Chem.*, xxi., 35), on solutions in methyl alcohol; and of Zanninovich-Tessarini (*Ibid.*, xix., 251), on solutions in formic acid.

The freezing-point method can also be successfully applied to only a limited number of solvents, and especially well only to water solutions. It may, perhaps, be applicable to solutions in benzene, acetic acid, and other solvents whose freezing-points are not widely removed from the ordinary temperature. But many solvents do not come within its range because their points of solidification lie too low to enable one to apply the freezing-point method with even a fair degree of accuracy. For such solvents it seems that the boiling-point method is the only one available, and this only when it has been worked out with sufficient accuracy to enable one to place a fair amount of confidence in the results. On the other hand, the boiling-point method is not easily applied to water solutions, owing in part to the very small value of the constant for that solvent.

One of the causes why the physical chemistry of to-day is primarily the physical chemistry of water solutions, is doubtless the greater dissociating action of that solvent. But another, which is strongly operative, is the absence of a reliable method with which to measure accurately the amount of dissociation in other solvents. This alone would show the importance attached to such determinations.

The interest in such measurements is, however, still further increased by the relation between the dielectric constants of solvents and their dissociating power, which has been pointed out on theoretical grounds for the first time in a readily accessible scientific journal, by J. J. Thomson (*Phil. Mag.*, xxvii., 320), and about the same time by W. Nernst (*Göttinger Nachrichten*, 1893, and *Zeit. Phys. Chem.*, xiii., 531). Whether such a relation exists rigidly and quantitatively, whether dissociating power is a function of any one physical property of the solvent, can be determined only by a careful study of the dissociation of electrolytes in the different solvents in question. In order that such work may be done, it is necessary that a method be worked out which will meet the requirements of practicability and accuracy.

The object of this preliminary notice is to call attention to the fact that we have such, in an improved form of the Beckmann boiling-point method, which he devised for the determination of the molecular weights of non-electrolytes. The essential feature in the new boiling-point apparatus (*Amer. Chem. Journ.*, xix., 581) is the introduction of a platinum cylinder around the thermometer, which cuts off the effect of radiation, and at the same time prevents the condensed solvent from coming in contact with the thermometer until it has been re-heated. The determinations of the molecular weights of a number of non-electrolytes in five solvents, whose boiling-points vary from 35° to 182°, show that we have to deal with the simplest efficient boiling-point apparatus thus far constructed. We have already applied it to the measurement of the electrolytic dissociation of solutions of two electrolytes, potassium iodide and sodium acetate, in ethyl alcohol, and the results will show the degree of accuracy of which the method is capable.

The reason why the boiling-point method has not

already found extensive application in measuring dissociation is, doubtless, to be sought in the fact that it has not been worked out with accuracy sufficient to enable it to be applied with a fair degree of probability.

The alcohol used in this work was dehydrated in the usual manner. It was allowed to stand for some days over quicklime, distilled and preserved in contact with anhydrous copper sulphate. The sodium acetate used was prepared by dissolving metallic sodium in water, neutralising the sodium hydroxide formed with acetic acid, evaporating the solution of sodium acetate to dryness, and finally drying in an air-bath, above 100°, to constant weight. The results given below are distinctly tentative, and solutions of both of the compounds in question will be worked over in the future with still greater care than has been possible thus far. The prime object in publishing these values at present is to show the fair agreement between the dissociation values as determined for the same substances, under the same conditions.

The Results.

SOLVENT, ALCOHOL. Molecular rise in 100 grms., 11.5°.

Potassium Iodide, molecular weight 166.

Grms. solvent.	Grms. potassium iodide.	Grms. iodide to 100 grms. solvent.	Rise in boiling-point.	Molecular rise.	Dissociation. Per cent.
53.553	1.6470	3.076	0.271°	14.625	27.2
56.234	1.2945	2.302	0.200	14.419	25.4
54.533	1.080	1.9804	0.172	14.417	25.4
58.572	1.389	2.371	0.208	14.566	26.7
58.900	1.060	1.800	0.158	14.576	26.7

Sodium Acetate, molecular weight 82.1.

Grms. solvent.	Grms. sodium acetate.	Grms. acetate to 100 grms. solvent.	Rise in boiling-point.	Molecular rise.	Dissociation. Per cent.
57.599	0.747	1.2969	0.185°	11.721	18
57.599	1.217	2.1129	0.299	11.616	10
57.878	0.5997	1.0361	0.147	11.648	13
57.878	0.9715	1.6785	0.238	11.640	12

In these determinations the barometer was read every few minutes, and the boiling-points were corrected for any change in atmospheric pressure, however slight. While very small changes in the barometer can be neglected when the question under investigation is one of molecular weights, they must always be carefully taken into account when far more accurate measurements are necessary, as in the case of the measurement of dissociation.

It will be seen from the results that the dissociation of potassium iodide in alcohol is between one-third and one-fourth of that in water at the same dilution. This is just about the same ratio as exists between the dielectric constants of these two solvents.

The dissociation found for sodium acetate in alcohol is almost certainly too low. Indeed the metallic sodium used in preparing the hydroxide was found to contain more than a trace of potassium, and this may account in part for the very low values.

We propose to extend the application of this method to a large number of electrolytes, and to as many solvents as may be practicable.

A Reception was held on Tuesday, the 24th inst., at the Galleries of the Royal Institute of Painters in Water Colours, Piccadilly, by Dr. and Mrs. Stevenson, Prof. and Mrs. Frank Clowes, and Dr. and Mrs. Bernard Dyer, representing the Institute of Chemistry of Great Britain and Ireland, the Society of Chemical Industry, and the Society of Public Analysts. The reception was very well attended, and a good programme of music was given, interspersed with humorous glees.

ON THE
SEPARATION OF THE OXIDES OF CERIUM
AND THORIUM.*

By G. WYROUBOFF and A. VERNEUIL.

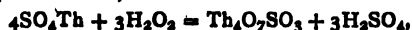
EVER since the commencement of our researches on the rare earths we have been endeavouring to find more certain, and above all more rational, methods of separation than those which have been in use up to the present time. All the known processes are in fact empirical; they do not depend on any definite or precise reaction, and, moreover, do not lead to any result except by a long series of fractionations. Further, we can never be sure of the purity of the substance obtained, except by spectroscopic observations, in the case when the spectrum is very characteristic, or by the absence of incandescence when dealing with thorina. Nevertheless all these earths, closely allied as they are, ought to have characteristic reactions; it is only under such conditions that they can be considered as distinct chemical species. The problem is thus reduced to finding these characteristic reactions, and to make the best use of them.

Let us first remark that, in placing the rare earths according to their chemical analogies, we obtain the following series:—Th, Ce, La, D, Yt (Er, Te). This series presents one extremely interesting peculiarity. If we succeed by any special process in completely separating one term of the series from the term immediately following it, we separate it, at the same time, from all those terms which follow, and leave all which precede it. It is due to this that all the known methods for separating the yttria earths from didymium carry with the latter not only the other cerite earths, but also thorina; it is equally for this reason that the reactions which enable us to obtain cerium, free from lanthanum and didymium, eliminate at the same time the yttria earths, but necessarily carry down thorina. We shall later on give a very simple explanation of this apparently singular phenomenon; but for the present it suffices to make the fact known, as it should serve as a basis for all rational methods for the separation of the rare earths. We are doubtless still far from possessing methods of separation for all the metals of the series, but for some of them this is already possible, and we now propose to describe the conditions of the reactions.

In a previous paper (*Bull. Soc. Chim.*, Series 3, vol. xvii., p. 679, 1897) we gave a very simple method for the complete separation of cerium from lanthanum and didymium. Conformably with the rule we have just laid down the yttria earths are eliminated at the same time, but also, conformably with this rule, the thorina remains with the cerium. It is necessary for us to obtain cerium rigorously free from lanthanum, didymium, and thorium; the problem is set in a very precise manner; the lanthanum and didymium, and the yttria earths, are all eliminated at the same time. We must now search for a reaction which will enable us to immediately separate the thorium. The solution of the problem stated in these terms does not present any difficulty. Phosphate of thorium is, in fact, quite insoluble in dilute hydrochloric acid; cerous phosphate is, on the other hand, easily soluble. If, therefore, to a mixture of chlorides containing a little thorina, we add enough phosphoric acid to saturate the latter, it separates integrally, carrying down with it, it is true, variable quantities (a few hundredths) of cerium. But in this form phosphate of thorium is an exceedingly voluminous body, very difficult to filter and wash. It is therefore necessary, after the addition of the phosphoric acid, to evaporate the whole, either on a water-bath or over a naked flame, to a pasty consistency; take up with HCl diluted with about seven or eight times its volume of water. The washing of the precipitate is then very easy,

and the filtrate does not contain a trace of thorium. To prove the absence of thorium a reagent is necessary, capable of showing even very minute quantities, and this we have in peroxide of hydrogen. It is in this manner that we have succeeded in solving not only the inverse problem,—the easy preparation of thorium free from cerium,—but also in effecting the quantitative separation of the two bodies.

M. Clève noticed, a long time back (*Bull. Soc. Chim.*, xliiii., p. 57, 1885), that on adding an excess of peroxide of hydrogen to a solution of sulphate of thorium, we obtain a voluminous white precipitate composed as follows:— $\text{Th}_4\text{O}_7\text{SO}_3$, (Th=116). This compound is not completely insoluble, and the filtrate gives a more or less abundant precipitate with ammonia. This is caused by the presence of free sulphuric acid which is formed during the reaction,—



and which tends to decompose the peroxide formed. It thus seemed probable that by using a less energetic acid we might succeed in completely precipitating the thorina, the more so as a similar reaction has long been known with cerium. We know, in fact, that acetate of cerium is completely precipitated in the state of peroxide by peroxide of hydrogen.

Experiment has fully confirmed this expectation; peroxide of hydrogen precipitates thorina integrally when it is in the state of chloride, and more especially when it is present as a neutral nitrate. The analogy between thorium and cerium is shown here in a striking manner. Both give peroxides in acid solution, by adding peroxide of hydrogen, and the difference depends only on the relative stabilities of these peroxides—that of cerium decomposes instantly in the presence of nitric or hydrochloric acid. It is on this very unequal stability of the two peroxides that we have endeavoured to found a method of separation. Theoretically the facts present themselves in a very simple fashion. If, to a mixture of acetates of Th, Ce, La, Di, and Yt, we add peroxide of hydrogen in excess, the thorium and cerium only are precipitated; the mixture Th and Ce, transformed into nitrate and treated again with peroxide of hydrogen, gives a precipitate having for its composition $\text{Th}_4\text{O}_7\text{N}_2\text{O}_5$,* the cerium remaining in solution. But when dealing with rare earths we should beware of jumping to conclusions, even those apparently the most legitimate; secondary circumstances intervene more often than would be believed, and create a veritable abyss between theory and its application.

The acetate of the peroxide of thorium is a body so gelatinous that it is impossible to filter it, and still more impossible to wash it. In the second place, the precipitation in acetic solution by means of H_2O_2 carries down, with the thorium and cerium, a small quantity of the other earths. This quantity diminishes, it is true, in proportion as the solution contains more free acetic acid; but the solubility of the cerium increases at the same time, and we thus lose in quantity what we gain in purity. An analogous phenomenon is produced when we attempt to separate thorium from cerium in nitric solution. The precipitate of the peroxide of thorium, which ought to be absolutely white, is always more or less yellow, and the more so as the quantity of cerium present in the mixture is greater. When this quantity is very small, all the thorina is not precipitated; there remains a certain quantity, very small it is true, in solution with the cerium. But in this case this double entanglement, which at first sight appears to be unfavourable, is, as a matter of fact, most favourable. So long as there remain traces of thorina mixed with the cerium, peroxide of hydrogen will

* Analysis gave us per cent:—

	Calculated.	Found.
ThO	100.0	100.0
O	9.09	9.13
N ₂ O ₅	20.45	20.45

* From the *Bull. Soc. Chim.*, Series 3, vol. xix.—xx., No. 6.

produce, in the solution of nitrates, a precipitate easily seen on account of its large volume, and, so long as the thorium contains ever so little cerium, the filtrate will give a flocculent yellow precipitate on the addition of ammonia. These reactions are so sensitive that 0.1 per cent of thorium in cerium, or cerium in thorium, can easily be precipitated and collected on a filter.

We are thus in possession of a reagent which not only enables us to recognise the presence of very small quantities of one of these earths in a solution of the other, but also to obtain them both perfectly pure.

Purification of Cerium.—As a rule cerium only contains small quantities of thorium. In such a case we add peroxide of hydrogen to the solution of the nitrates, which has previously been evaporated to dryness or rendered neutral with ammonia, and we then heat to boiling for a few minutes. A small quantity of the filtered solution should not give any precipitate when treated with its own volume of peroxide of hydrogen and boiled again. The yellow precipitate, in the form of a voluminous yellow mass, must not be confounded with the whitish turbidity obtained by the action of peroxide of hydrogen when warmed on pure cerium; this turbidity is caused by the phosphoric acid which is always present in commercial peroxide of hydrogen, which forms an insoluble cerous phosphate, especially when heated. Cerium thus treated does not contain any trace of thorium. If the amount of thorium mixed with the cerium is at all considerable, —15 to 20 per cent, for example,—it will be of advantage to previously remove the greater part of it by means of carbonate of ammonium and ammonia. The thorina is easily soluble, and only carries a few hundredths of cerium with it. After a single treatment only, the cerium remaining insoluble contains no more than 5 or 6 per cent of ThO; it is then neutralised with HNO₃, and precipitated with peroxide of hydrogen. What we have just described for the treatment of cerium applies equally well to a mixture containing all the earths of cerite and yttria.

Purification of Thorina.—Here it is the inverse reaction that is produced; thorina is nearly always accompanied by large quantities of foreign earths, about 95 per cent in monasite.

The method to be followed depends on what we intend to do. If we wish to extract the whole of thorina existing in the mixture, it is best to treat the solution of the nitrates by peroxide of hydrogen (about 10 or 15 c.c. is necessary, supposing it to be in the proportion of 10 vols. per grm. of ThO). The thorina thus precipitated is very impure; white at first, it rapidly becomes yellow, and may even take a deep orange colour. To purify it, it is necessary to re-dissolve it in warm nitric acid, evaporate to dryness, and re-precipitate with peroxide of hydrogen. It is then almost pure, though it still gives a slight hazy incandescence, comparable with a mixture containing 0.1 or 0.2 per cent of cerium. A third precipitation makes it perfectly pure. Its purity is proved in a very simple manner; the filtrate should not give the slightest precipitate with ammonia.

In the absence of cerium, the precipitation of the thorina by peroxide of hydrogen is, in fact, complete. If we wish to work rapidly, it is of advantage to leach the nitrates several times with a neutral solution of carbonate of ammonia, the solution evaporated to dryness only needs two treatments with peroxide of hydrogen to give a thorina without a trace of incandescence.

It is necessary to remark that the impurities which are to be found in commercial peroxide of hydrogen, especially phosphoric and sulphuric acids, accumulate in the peroxide of thorium. It is therefore necessary either to distil the peroxide of hydrogen used, an operation which is not difficult by following M. Hanriot's method (*Bull. Soc. Chim.*, New Series, xliii, p. 468, 1885), or else to purify the thorium obtained. This purification is not so easy as might be believed. It is best to dissolve it in HCl, and precipitate it by oxalic acid in a strongly acid

solution. We thus remove the greater part of the phosphoric acid, but not the sulphuric acid. Oxalate of thorium is unfortunately quite insoluble in boiling nitric acid: this distinguishes it from the oxalates of all the other rare earths; but, on the other hand, if it has not been previously warmed, it is instantaneously decomposed by cold caustic potash or soda, a reaction which is characteristic of it. The hydroxide thus obtained does not contain a trace of sulphuric acid. To get rid of the phosphoric acid still present, it must be re-dissolved in HCl and re-precipitated with oxalic acid, and the oxalate decomposed by soda.

The hydroxide thus obtained contains notable quantities of alkali, which cannot be got rid of by washing. It is therefore necessary to re-dissolve it in HCl, and to precipitate by ammonia. Such a hydroxide ought to be white after strong calcination, when obtained from the oxalate, nitrate, or sulphate. This degree of purity is only obtained with difficulty, for even the least traces of impurities contained in the reagents used give the calcined hydroxide a greyish or a yellowish colour, as mentioned in all previous works.

(To be continued).

PROCEEDINGS OF SOCIETIES.

PHYSICAL SOCIETY.

Ordinary Meeting, May 13th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

A paper by Prof. W. E. AYRTON and Mr. T. MATHER, on "*Galvanometers*," was read by Prof. Ayrton. It is a sequel to *Proc. Physical Soc.*, vol. x., p. 393, and to *Phil. Mag.*, vol. xxx., p. 58.

The authors suggest that in future the comparative sensitiveness of galvanometers should be expressed in terms of the number of millimetre scale-divisions per microampère, when the observed image or "spot" is 1 metre from the mirror. Unit angular deflection is therefore 1/2000th of a radian. Further, for the periodic time,—i. e., the time between two transits of the "spot" across some fixed point on the scale, in the same direction,—the standard should be ten seconds. It is also proposed to reduce the factor of sensitiveness, as regards resistance, to the common basis of 1 ohm. The assumption is that, for a given galvanometer, the deflection per microampère is proportional to the two-fifth power of the resistance of the windings. Tables accompanying the paper give complete data for a large number of galvanometers constructed during the past ten years, and it is possible to trace the improvements in sensitiveness throughout that time. The most sensitive galvanometers are the oscillographs; they have very short periods, the moving parts are small, the controlling fields very strong. They are designed to indicate the character of rapidly-varying currents. An oscillograph, as improved by Mr. Duddell, was exhibited; its period is 0.001 sec., and its factor of sensitiveness, according to the authors' classification, is greater than any yet obtained. A distinction is drawn as to the use of the term "dead-beat." Maxwell applies it to galvanometers in which the motion is "aperiodic," i. e., to those in which the suspended system, before coming to rest, passes only *once* through the position of equilibrium. This meaning is retained; it is not to be confused with "quick-moving" or "short-period." A pendulum illustrating these distinctions was exhibited. As regards insulation of galvanometers and shunt-boxes, the authors now apply the "guard-wire" principle of Mr. W. A. Price. The instrument to be insulated is enclosed in a metal case provided with a

terminal to which one end of the windings is connected. The second end of the windings passes out through an ebonite bush-piece. This arrangement is said to nullify leakage and to prevent electrostatic disturbance of the suspended system. In the second section of the paper, the authors calculate the limiting sensitiveness of galvanometers of the "Thomson" type. The investigation is based upon Prof. Schuster's B.A. 1894 paper; it takes into account the period of the suspended system and the specific magnetisation of the needle. Lastly, the authors discuss the relative merits of long and short periods, *i. e.*, the best "control," for galvanometers intended to indicate zero points in potentiometer operations. They conclude that if the control can be readily altered, and if the sensitiveness can be adjusted for the test, then, for rapidity of working, the "control" should be so adjusted that the sensitiveness is approximately two or three times greater than is absolutely needed for the desired accuracy.

Prof. THRELFALL thought the authors' method of comparing galvanometers very misleading. The results obtained in their comparison of the oscillograph (3,310,000) and the suspended-coil galvanometer (27) might be regarded as the *reductio ad absurdum* of the proposed system. The absurdity arose from the dissimilarity of the two instruments. Moreover, the proposed system ignored the fact that sensitiveness may be obtained by optical as well as by electro-magnetic means. Optical sensitiveness, owing to its greater stability, was to be preferred to electro-magnetic sensitiveness. The fundamental problem in the construction of galvanometers is an optical one; it is necessary to decide the mass and dimensions of the suspended parts so as to ensure (1) optical accuracy, and (2) electro-magnetic sensitiveness. Thus, to some extent, the weight of the mirror determines the thickness of the suspension. As an instance of what might be done by optical methods, Prof. Threlfall referred to work done by himself and Mr. Brearley (*Phil. Mag.*, 1896), in which it was possible to measure to 1.48×10^{-12} amperes, and, with special refinements, to 3×10^{-14} amperes. He had found that the best diameter for glass mirrors was 1.1 c.m., with a weight just under 0.5 gm. These were used with a scale at 276 c.m., read by a microscope to 0.04 m.m. The course of the light was:—Lamp, large lens, small scale, mirror, eyepiece. The period was 25 secs., and the resistance 50,000 ohms. Even better results could be obtained by using mirrors of quartz or of blood-stone. Quartz is incomparably to be preferred to glass. Such figures indicated what could be done by optical sensitiveness—the sensitiveness that the authors ignored. It was pointed out by Prof. Threlfall that the controlling field for galvanometers of the "Thomson" type should be straight and uniform. This was best secured by using two magnets, one above and one below the needles.

Prof. PERRY said the authors had not asserted that a galvanometer with higher figure of merit, according to their classification, was *superior* to another of lower figure. It must be agreed that the figure they obtain is a very valuable datum for the comparison of instruments designed for similar purposes, for instance in classifying those used by Prof. Threlfall. Mr. Duddell was to be congratulated on the extreme sensitiveness and small period of his oscillograph.

Prof. AYRTON, referring to Prof. Threlfall's *reductio ad absurdum*, admitted that the criticism would carry some conviction if the two instruments were of different kinds; if, for instance, one possessed a suspended needle and the other a suspended coil. But the argument failed, because both instrument were of the suspended-coil type. In one of them Mr. Duddell had developed the advantages to be gained by reducing the air-gap. To form an opinion of electro-magnetic improvements in galvanometers, it was necessary to reduce the results of all instruments to some system of classification. There was no objection, after that, to adding a good mirror, and reading by a good microscope.

The PRESIDENT proposed votes of thanks to the authors, and the meeting adjourned until May 27th.

NOTICES OF BOOKS.

Text-book of Physical Chemistry. By C. L. SPEYERS. New York: D. van Nostrand and Co. London: E. and F. N. Spon.

THE subject-matter of this book is almost entirely confined to the relations between the various forms of energy manifested by chemical reactions and physical phenomena.

The author considers that energy exists in five principal forms, *viz.*, mechanical, heat, electrical, magnetic, and radiant energy, with several subdivisions. He gives no place to chemical energy, as he considers this can always be identified under one or other of the above forms, and we are inclined to think he is right in so doing. His speculations—for they are nothing more—on the ultimate constitution of matter and energy are not very convincing; he apparently wishes to dispense with matter altogether, and substitute energy in its place, but, unless he attaches two separate meanings to the word energy, we cannot see that his ultimate result has any meaning. On page 2 he defines the different forms of matter as "collections of forms of energy in space."

On page 3 he states that "we need energy and a something to enable energy to collect in space before we get a material substance. This something which enables, and perhaps causes, the energy to collect in space we shall call matter."

The combination of these two leads to the puzzling statement that "We need energy and a collection of forms of energy in space, to enable energy to collect in space, before we get a material substance." We must confess that this is too deep for us.

The author, however, has not attempted to carry these speculations into the body of the book,—the persistent use of the expression "forms of energy," instead of matter, being the only indication that he has novel ideas with regard to the phenomena he is discussing.

The equations expressing the relations of energy in its various forms are well worked out from first principles in the usual manner, and many problems exemplifying their use are interspersed throughout the work.

With the exception of the first chapter, which is in places unintelligible, and the second, which is debatable, we can thoroughly recommend this work to the class of students for whom it is written.

Notes on Observations. By S. LUPTON. Macmillan and Co.

THIS little book is intended to assist young physicists in realising the value of the results which they have obtained in physical and chemical experiments. The first six chapters, being mainly metaphysical, are rather outside the ostensible scope of the book, and call for no comment.

The rest of the book is devoted to the mathematics involved in computing the value of a series of observations, and in recording and deducing results from them. The author hardly does justice to the graphical method of recording results. Of course the curve should never be used for scaling intermediate values, if any but the roughest approximation is required.

The chapters on interpolation, and the treatment of abnormal or doubtful results, are useful, the latter especially deserving careful attention. The principles involved in the construction of empirical formulae are well set out in the final chapters.

On the whole we should say that, although it is in many ways unlikely to be of immediate use to beginners in experimental science, it is likely to be of great value in preparing them for what will be expected of them when they enter upon the serious business of life.

The Proceedings of the Chemical and Metallurgical Society of South Africa. May, 1894, to January, 1897. Vol. I. Edinburgh: R. W. Hunter. Johannesburg, S.A.R.: 12, Chamber of Mines Buildings.

We are pleased to welcome the first volume of "The Proceedings of the Chemical and Metallurgical Society of South Africa," comprising work done during the first three years of its existence. The subjects generally brought before the Society naturally deal mostly with gold-mining, smelting, assaying, &c.

The development of the cyanide process has attracted a good deal of attention, and many interesting and valuable papers, followed by not less valuable discussions, are to be found in these pages.

We will not here go into any further details of the work of the Society, as the reports of the monthly meetings are generally abstracted in another column as they arrive. We will conclude by offering our hearty congratulations to the Society for the plucky manner in which it keeps going ahead under most disadvantageous circumstances, hindered and trammelled as its work must be by the most obstructive Government of modern times.

The Elements of Electro-chemistry treated Experimentally. By Dr. ROBERT LÜPKEN. Translated by M. M. PATTERSON MUIR, M.A. With 54 Figures in the Text. London: H. Grevel and Co. 1897. Pp. 223.

Of late years electricity has been making great strides in every direction, and it is now being applied more and more to chemical work; but to carry out this work in a satisfactory manner, it is first necessary for the student to be thoroughly grounded in the subject of electro-chemistry or electrolysis.

Part I. of this book is on recent theories of electrolysis. Hittorff's conception of electrolytes was that "they are salts, and are separated by electrolysis into the same atoms, or atomic groups, as the exchange in their chemical reaction with one another." All other substances, whether liquids or dissolved salts, are non-conductors. It occurred to Faraday to send the same current through several cells in series, each containing a different electrolyte; the result of this experiment was expressed in his "law of constant electrolytic action." In 1881 H. von Helmholtz, in the Faraday lecture, stated that "every single valency of an elementary or compound ion is charged with exactly the same quantity of positive or negative electricity, which behaves as if it were an electrical atom that cannot be further divided."

Part II. deals with the theory of solutions of van't Hoff, osmotic pressure, the vapour pressure of solutions, the boiling and freezing points of solutions, &c. The osmotic theory has been of great service in the practical work of chemistry, as it has called into existence very valuable methods for determining molecular weights. In chapter I of this part experiments illustrating osmotic pressures are described, the results of which lead to Pfeffer's law that "The osmotic pressure of a solution is proportional to the concentration and also to the absolute temperature."

In Part III. we come to the osmotic theory of the current of galvanic cells; here the theory of Nernst, based on the propositions laid down in Parts I. and II., of the production of the electric current in galvanic cells and the deductions made from it are considered. Nernst explains the occurrence of a difference of potential between solutions of different concentrations of the same electrolyte,

by the different velocities of the ions set in motion by osmotic pressure, by which means the cations become in excess in one solution and the anions in the other; these are called liquid-cells; a concentration-cell consists of two bars of the same metal, placed in solutions of a salt of the metal of different concentrations, these solutions being in contact; a current is produced until the concentrations become equalised. Reduction- and oxidation-cells are again different, and are dependent for their action on chemical energy, which is transformed into electrical energy. Chap. 5 of Part III. is on the solution-pressure of the metals, and to arrange the metals in their order of solution-pressure it is only necessary to determine whether one metal is able to precipitate another from a solution of one of its salts, and thus, because of its greater solution-pressure, draw away the electrical charges to the cations of the second metals.

A few chapters on intensity of fixation and polarisation, irreversible cells, accumulators, and the energetics of galvanic elements, close a most interesting and clearly written volume, on which the author is to be congratulated.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 15, April 12, 1898.

The President announced the death of M. Aimé Girard, Member of the Section of Rural Economy. The meeting was adjourned in consequence.

Observations relative to the Action of Oxygen on Sulphide of Carbon and to the Chemical Influence of Light. Preliminary Work to Determine the Reactions.—M. Berthelot.—After some introductory speculative remarks the author gives a general idea of his experiments. A sealed tube containing about 1 gm. of sulphide of carbon is placed in a flask of 2.5 to 3 litres capacity, large enough for the vapourised sulphide of carbon to fill at a temperature of 15°. The neck of the flask was then contracted and sealed, and the small tube broken by careful shaking. Three such flasks were prepared; two were left on the laboratory table in bright light, but away from direct sunlight; the third was placed unsheltered on the roof. The experiment lasted 379 days, when the flasks were examined. The first contained 1.055 gm. of CS₂; the glass was quite clear, and no sulphur was deposited; no carbonic acid was found, but there was a little gas present which turned out to consist of O = 20.7 and N = 79.3. The second flask (also kept in the laboratory) behaved similarly, but the residual gas had the composition O = 20.9 and N = 79.1, but in neither case had the CS₂ undergone any change. The third flask, on the roof, containing 1.07 gm. of sulphide of carbon exposed to all weathers and direct sunlight, was quickly acted upon; after only a few hours a white or yellowish deposit formed on the interior surface of the glass, and this increased progressively; at the end of the year the gas was found to consist of—

	Per cent.
CO ₂	1.5
CS ₂	8.5
CO	2.3
O	14.7
N	72.0
Water vapour	1.0

100.00

The solid matter was found to consist of—

	Grm.
Sublimed sulphur	0.104
Carbonaceous matter.. .. .	0.030
Carbonic acid	0.0024
Sulphide of carbon and unestimated matter	0.0136

Absorption of Oxygen by Pyrogallate of Potash.—M. Berthelot.—Experiments were carried out on solutions of widely varying strengths of pyrogallol and of potash, and even with solid potash, but always according to exact equivalents, from $\frac{1}{4}$ equivalent of potash up to 3, and further from the ordinary up to high temperatures. The paper is too long to usefully abstract in a short space.

Rays Emitted by Compounds of Uranium and of Thorium.—M^{me}. S. Curie.—Two plates were arranged as a condenser, one of them being covered with a uniform layer of uranium or other substance finely powdered. A difference of potential of 100 volts was established between the two plates, which were kept at 3 c.m. apart; a large number of metals, salts, oxides, and minerals were experimented on to find out if any of them had, like uranium, the power of making air a conductor of electricity. This was found to be the case. It was noticed that the more uranium present in any of its salts, the more active that salt was; the compounds of thorium are very active, while oxide of thorium surpasses that of even metallic uranium. It is noticeable that uranium and thorium are, of the elements tried, both the most active and also have the highest atomic weights. Good photographic impressions have been obtained with uranium, uranous oxide, pitchblende, chalcocite, and oxide of thorium. These bodies will act through air, glass, or aluminium.

The Combinations of Pyridine and of Trimethylamine with Formic and Acetic Acids.—G. André.—The author has followed up his researches on the compounds formed by formic, acetic, or propionic acids with pyridine. He has now gone further, and finds that the combinations between trimethylamine and formic and acetic acids are much more stable than those formed by pyridine with the same acids. The formic compound has the same type of formula as that obtained with pyridine, but the acetic compound is much richer in acid. These two combinations with trimethylamine both have a notably higher boiling-point than either of their components.

Bulletin de la Société Chimique de Paris.

Series 3, Vol. xix.-xx., No. 4.

Separation and Estimation of Lead, Copper, and Arsenic.—F. Jean.—Already inserted.

Some New Derivatives of Homopyrocatechin.—H. Cousin.—Homopyrocatechin has been but little studied up to the present time, and, with the exception of a few ethers, none of its derivatives are known. By adding on 1 molecule of homopyrocatechin with 1 molecule of iodide of methyl in the presence of potash, a mixture of cresol, $\text{CH}_3\text{—C}_6\text{H}_3\text{—OCH}_3\text{—OH}$, and its isomer,

$\text{CH}_3\text{—C}_6\text{H}_3\text{—OH—OCH}_3$, was obtained. Iodide of

methyl in excess gives homoveratrol, $\text{C}_7\text{H}_5\text{—(OCH}_3)_2$. Iodide of ethyl gave analogous results. By treating homopyrocatechin in acetic solution with a current of chlorine, two chlorinised bodies were obtained:— $\text{C}_7\text{H}_5\text{Cl}_2\text{O}_2 + 2\text{H}_2\text{O}$, in white needles, losing its two molecules of water *in vacuo*, and melting at $179\text{—}180^\circ$; and chlorinised methylorthoquinone, $\text{C}_7\text{H}_3\text{Cl}_2\text{O}_2$, crystallising in small bright red microscopic plates slightly soluble in alcohol, more so in acetic acid, and melting at $97\text{—}98^\circ$. Reducing agents change this substance to $\text{C}_7\text{H}_5\text{Cl}_2\text{O}_2$. Analogous bodies are formed by the action of bromine.

The author has also isolated two mononitrated homopyrocatechines and the corresponding homoveratrols. 5-Nitrohomopyrocatechin, $\text{C}_7\text{H}_4\text{O}_2\text{—NO}_2$; 5-nitrohomoveratrol, $\text{C}_7\text{H}_5\text{—(OCH}_3)_2\text{—NO}_2$; and 6-nitrohomopyrocatechin, $\text{C}_7\text{H}_5\text{—(OH)—NO}_2$; 6-nitrohomoveratrol, $\text{C}_7\text{H}_5\text{—(OCH}_3)_2\text{—NO}_2$. The action of sulphuric acid on homopyrocatechin gave only one sulphur derivative, viz., homopyrocatechin monosulphonic acid, $\text{C}_7\text{H}_5\text{—(OH)—SO}_3\text{H}$. It is a deliquescent crystalline mass, very easily soluble, melting at $93\text{—}94^\circ$. The potash salt crystallises in fine needles, and is soluble in water and in boiling alcohol. Heated in a sealed tube with iodide of methyl and alcoholic potash, it gives a homoveratrol sulphonate of potassium, $\text{C}_7\text{H}_5\text{—(OCH}_3)_2\text{—SO}_3\text{K}$.

Zeitschrift für Analytische Chemie.
Vol. xxxvii., No. 2.

Some Contributions to the Chemistry of Proteid Precipitates.—H. Schjerner.—The author examines the proteid precipitates obtained from beer, Merck's diastase, Witte's peptone, egg albumen, and milk. He uses chlorides, acetates, and sulphates of the metals Pb, Cu, Hg, Mg, Fe, Ca, Sr, Ba, Zn, Co, and Mn, and obtains precipitates, which are washed with water and weighed. He is unable to obtain precipitates of the noble metals quantitatively, probably owing to the ease with which the oxide is reduced to the free metal.

Method of Determining the Fineness of Meal.—Dr. Višor Vedral.—It has already been shown that the fineness of meal is proportional to the ash content, and the author gives a simple method of determining this ash content. He takes six crucibles containing about 5 grms. of meal and heats them for 6 to 8 hours in a Fletcher's muffle oven, cools them in a desiccator, and weighs again. By adding a little HCl and extracting with water the ash can be examined and estimated.

Colorimetric Determination of the Weight of Smoke.—Dr. P. Fritzsche.—This is usually determined by allowing a measured volume of the gas from the chimney to pass through a glass tube prepared with asbestos, and estimating the obtained carbon as CO_2 . This method, however, is lengthy, and has to be done in a laboratory. The author has devised a simple method, in which a tube is prepared with loose cellulose to collect the soot. This is connected on one side with another tube, which can be inserted in the chimney, and on the other side with an aspirator. About 10 to 20 litres of gas are drawn in by the aspirator. The cellulose and soot are placed in a flask with 200 c.c. of water and well shaken till the colour is a uniform grey. This is compared with standard colours obtained by shaking up the same quantity of cellulose with 5, 10, 15, 20, 25, and 30 grms. of soot in 200 c.c. water. After a little experience these soot determinations can be performed accurately in a few minutes.

Investigation of Commercial Incandescent Bodies.

—E. Hintz.—The author analyses 40 specimens, and finds that they all consist chiefly of thorium, with a small percentage of cerium oxide and varying traces of neodymium, yttrium, zirconium, lime, magnesia, and silica. He then investigates photometrically incandescent substances containing known proportions of the various earths, and comes to the conclusion that if they contain approximately 99 parts of thorium to 1 part of cerium, oxides of neodymium, lanthanum, and yttrium up to 1 per cent do not influence the lighting-power, while the influence of 0.1 per cent of zirconium is uncertain.

MISCELLANEOUS.

Royal Institution.—On Tuesday next, May 31st, Professor S. H. Butcher begins a course of two lectures at the Royal Institution, on "Literary Criticism in Greece"; on Thursday, June 2nd, Dr. Edward E. Klein delivers the first of two lectures on "Modern Methods and their Achievements in Bacteriology"; and on Saturday, June 4th, Dr. Richard Caton begins a course of two lectures on "The Temples and Ritual of Asklepios at Epidaurus and Athens" (with lantern illustrations). The Friday Evening Discourse on June 3rd is by Professor W. M. Flinders Petrie, on "The Development of the Tomb in Egypt"; that on June 10th is by Lord Rayleigh, whose subject is "Some Experiments with the Telephone."

On the Assay of Minium.—At the Second International Congress of Applied Chemistry, M. Forestier made the following communication in view of the adoption of a uniform method of commercial assays. According to him, the value of a minium depends on its percentage of pure minium, Pb_3O_4 , and the percentage of impurities. Forestier proposes to estimate the latter in the following manner:—10 grms. of minium are treated in a flask, with 10 grms. of ordinary sugar dissolved in 50 or 60 c.c. of boiling water, and 10 c.c. of nitric acid at 36°, until the red colour has entirely disappeared. If there is much residue the mixture must be heated for a quarter of an hour. The insoluble matter is collected on a tared filter and weighed. To estimate the Pb_3O_4 , the author treated the 1 gm. of the substance with acetic acid; this decomposes the oxide, leaving a residue of PbO_2 ; he used 10 c.c. of acid at 10°, and 20 c.c. of water, heated for half an hour on the water-bath, and collected the binocide. The weight of this is determined either by direct weighing or titrimetrically by the iodine process. It should be noticed that the author replaces the nitric acid generally used by acetic acid; according to him the former dissolves a part of the binocide. The Congress admitted the Forestier process.—*Zeit. f. Ang. Chem.*, 1898, No. 11, p. 176.

MEETINGS FOR THE WEEK.

TUESDAY, 31st.—Royal Institution, 3. "Literary Criticism in Greece," by Professor S. H. Butcher, LL.D.

THURSDAY, June 2nd.—Royal Institution, 3. "Modern Methods and their Achievements in Bacteriology," by Edward E. Klein, M.D., F.R.S.

Chemical, 8. "The Action of Ether on Organic Acids and on Carbohydrates in presence of Hydrogen Bromide," by H. J. H. Fenton, M.A., and Mildred Gostling, B.Sc.

FRIDAY, 3rd.—Royal Institution, 9. "The Development of the Tomb in Egypt," by Professor W. M. Flinders Petrie, D.C.L.

SATURDAY, 4th.—Royal Institution, 3. "The Temples and Ritual of Asklepios at Epidaurus and Athens," by Richard Caton, M.D., F.R.C.P.

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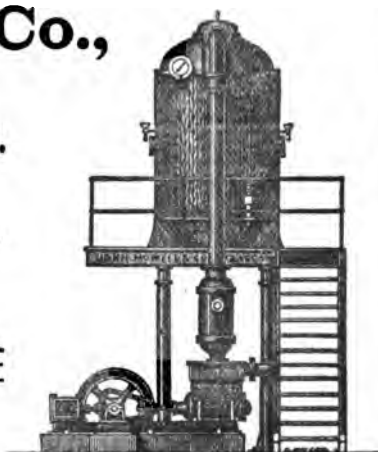
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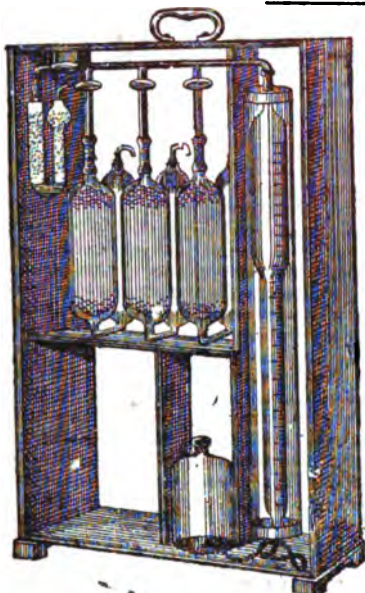
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CONTENTS.

ARTICLES:—	PAGE
On Atom Forms, as deduced from the Crystalline Modifications of the Elements, by Wm. L. T. Addison, B.A., M.B.	251
On the Separation of the Oxides of Cerium and Thorium, by G. Wyruboff and A. Verneuil	254
The Estimation of Manganese as the Sulphate and as the Oxide, by F. A. Gooch and Martha Austin	255
PROCEEDINGS OF SOCIETIES:—	
CHEMICAL SOCIETY—The Liquefaction of Hydrogen and Helium—The Action of Formaldehyde on Amines of the Naphthalene Series—On the Constitution of Oleic Acid and its Derivatives—Stereoisomeric Derivatives of Camphor	257
PHYSICAL SOCIETY—A Simple Method of Reducing Prismatic Spectra—&c.....	260
Density and Boiling-point of Liquid Hydrogen.....	261
OBITUARY—Lord Playfair	261
CHEMICAL NOTICES FROM FOREIGN SOURCES	261
MEETINGS FOR THE WEEK	262

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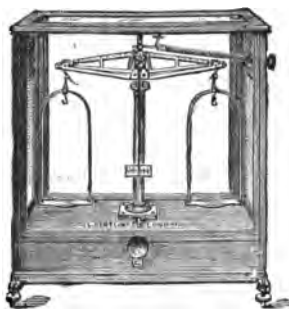
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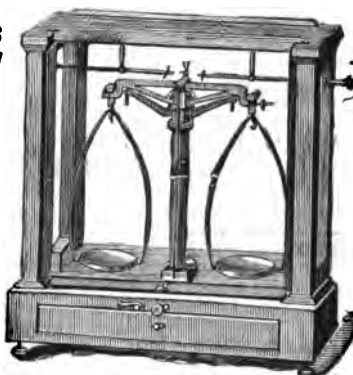


FIG. 34.

SHORT BEAM BALANCE, No. 108, Fig. 34, to carry 100 grammes in each pan, and turn to 1.10th milligram. Steel knife-edges, agate planes, two rider apparatus, and new improved arrangement for arrest of pans

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THE CHEMICAL NEWS.

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ON ATOM FORMS, AS DEDUCED FROM THE CRYSTALLINE MODIFICATIONS OF THE ELEMENTS.*

By WM. L. T. ADDISON, B.A., M.B.

THE relationships outlined in this paper are too large and many to be fully discussed in the space allotted.

That such a difficulty may be somewhat met it was thought advisable to treat the greater portion of the subject in outline merely, stating as facts such as may be demonstrated by observation or geometric proof, and to discuss at greater length the simplest of crystalline allotropic elements—phosphorus.

In the study of crystallography, one's attention reverts to those substances which are most common and most crystalline in character. Of such substances, calcite has been selected by observer after observer, from the time of Nicholas Steno down to the present. Among the workers in this science was Mitscherlich, who observed that similar chemical compounds had the same form and could intercrystallise in the same crystal. Thus, calcium, magnesium, manganese, ferrous and zinc carbonates crystallise and intercrystallise in rhombohedra of approximately the same proportions. Thus, form is consequent on the chemical grouping, and is a function of that group of atoms.

There is in the carbonate group a central atom carbon, about which the other atoms are grouped; and these outer atoms are the outer portions of the molecule, or those of easiest and first contact.

The grouping is at four attractive places, and the form of the crystal being constant, the form of the molecule and the position of these areas of attraction must also be constant.

Of carbon we may know much. It occurs in crystalline form, as the diamond, sp. gr. 3.6; and as graphite, sp. gr. 1.8 when pure, or one-half the specific gravity of the diamond.

The cleavage of the diamond is in regular octahedra; that is, the line of union of the carbon atoms lie in octahedral planes. This form may be given either by octahedra being placed edge to edge, or by tetrahedra similarly placed, or by an arrangement of the two together, in which they would fill space. It could not be an arrangement of both forms, and so must be of one of the forms. The tetrahedron has the same number of solid angles or prominences and the same number of faces as the carbon atom has areas of valence. In the octahedron, the differentiated portions are six solid angles and eight faces. Thus the differentiated portions of the tetrahedron are coincident in number with the chemical attractions of the carbon atom. If the carbon atom be a regular tetrahedron, then—in the arrangement described—the solid angles are the differentiated portions in contact, and they may be regarded as the areas of attraction.

If a series be taken in a diagonal axis of symmetry of the octahedral grouping, it is composed of two hexagonal series of regular tetrahedra, with their apices pointing in opposite directions. If one series be taken away, the atoms of the remaining series will be left with their solid angles only in contact; the specific gravity will be half that of the diamond or octahedral grouping, and the arrangement will account for a rhombohedral, a hexagonal,

and a gross form simulating a monoclinic one, the basal plane of which is at approximately $70^\circ +$ to the oblique axis; it also will account for the intermolecular mobility of graphite, and the triangular markings on the basal plane of that mineral.

Silicon, germanium, lead, and thorium occur in regular octahedra, as does carbon.

Tin, titanium, and zirconium dioxides are isomorphous, and tin is known to occur in quadratic octahedra, and may be accounted for as a grouping of quadratic tetrahedra.

Zirconium has been observed in tin-white microscopic monoclinic leaflets. Such leaflets occur in series parallel to P faces of the octahedra described for tin.

If the prominences or solid angles of the outline forms of all atoms be their areas of valence, as in carbon, phosphorus requires an outline form of five solid angles. Such a form is found in the bi-pyramidal hexahedron. Phosphorus occurs in two crystalline forms—1st, as regular octahedra and rhombic dodecahedra, of sp. gr. 1.826 to 2.089; and 2nd, as rhombohedra, of sp. gr. 2.34. That there is a regular form is a significant fact, which leads one to expect some regular characteristics in the primal form.

If the solid angles of the bi-pyramidal hexahedron (and we will in future refer to the outline form of the atom as a solid one) be arranged with three equidistant solid angles in a zone, and two apical solid angles, one upon either side of, and equidistant to, the plane of the zone, and at the same distances from the zonal angles, as the zonal angles are apart *inter se*, then a form is given composed of two regular tetrahedra, base to base, and of regular characteristics.

Such a form may be arranged with its edges together in two ways:—1st, With two apical solid angles opposite and symmetrically three zonal solid angles about the point of contact to give a rhombohedral form; and, 2nd, with eight apical solid angles about a point, and the zonal angles in pairs.

In the first arrangement, the angle R is $82^\circ 18' +$. In a continuous arrangement, columnar interatomic spaces are observed. These are as of regular octahedral spaces face to face. Thus, the upper and lower faces of these hypothetical octahedra are in apposition with those of the octahedra above and below. The remaining six faces are in apposition with the faces of the hexahedra, and each hexahedron has all its faces in apposition with those of octahedra; hence the proportion in number of octahedra to hexahedra is as the faces of each form in apposition with those of the other, or as one to one.

A regular octahedron of 2 inch edge is composed of two pyramids of base area 4 square inches and of $\sqrt{2}$ inches height, so that by the formula—

The solid contents of a pyramid = $\frac{\text{Area of base} \times \text{height}}{3}$

its solid contents are—

$$\left(\frac{4}{3} \times \sqrt{2}\right) 2 = 8 \frac{\sqrt{2}}{3} \text{ cubic inches.}$$

An octahedron of 4 inches edge has a cubic area—

$$\left(\frac{16}{3} \times \frac{2\sqrt{2}}{1}\right) 2 = \frac{64\sqrt{2}}{3} \text{ cubic inches.}$$

Such an octahedron is composed of six octahedra and eight tetrahedra of 2 inch edge. The six smaller octahedra have an area of—

$$\frac{48\sqrt{2}}{3} \text{ cubic inches,}$$

therefore the remaining eight tetrahedra have an area—

$$\frac{16\sqrt{2}}{3} \text{ cubic inches,}$$

* Abstract of a Paper read before the British Association (Section B), Toronto Meeting, 1897.

and each tetrahedron has an area—

$$\frac{2\sqrt{2}}{3} \text{ cubic inches.}$$

The hexahedron has an area of two tetrahedra, or—

$$\frac{4\sqrt{2}}{3} \text{ cubic inches,}$$

and the area occupied per hexahedron in the rhombohedral arrangement is its own area + that of an octahedron = $\frac{12\sqrt{2}}{3} = 5.6568$ cubic inches.

In the second or regular arrangement, in which eight apical solid angles meet about a point, the remaining apical solid angles form the solid angles of an outline cube, and the two opposite solid angles of the cube are apart—twice the distance between the apices of the hexahedron. If x = the edge of the cube, then the diagonal of its face is $x\sqrt{2}$, and the line between the two opposite solid angles = $\sqrt{(\text{edge})^2 + (\text{diagonal of face})^2} = x\sqrt{3}$; therefore, $x\sqrt{3} = 2$ (length of the hexahedron).

The length of the hexahedron may be found as follows:—The zone of the hexahedron is an equilateral triangle of 2 in edge. Bisect each side, and join the points of bisection to the opposite angles, thus bisecting the angle 60° . There are then six triangles, each one-half of an equilateral triangle of sides, y ; y = hypotenuse, or the line from the centre of the zone to the zonal angle. The other sides of the triangle are x and $\frac{1}{2}y$, so—

$$x + \frac{1}{2}y = y \text{ and } \frac{2}{3}y = x, y = \frac{2}{\sqrt{3}}$$

On the triangle of the line y , the line from the centre of the zone to the apex of the hexahedron, and the edge of the hexahedron joining these lines, let z = the line dropped from the apex of the hexahedron to the centre of its zone, then—

$$x^2 + y^2 = 4, x^2 + \frac{1}{4}y^2 = 4, x^2 = \frac{15}{4} \text{ and } x = \frac{2\sqrt{2}}{\sqrt{3}}$$

and the length of the hexahedron is—

$$\left(\frac{2\sqrt{2}}{3}\right)2 = \frac{4\sqrt{2}}{\sqrt{3}} \text{ inches.}$$

In the cube of edge x ,—

$$x\sqrt{3} = \frac{8\sqrt{2}}{\sqrt{3}} x = \frac{8\sqrt{2}}{3},$$

and 8 hexahedra occupy—

$$x^3 = \left(\frac{8\sqrt{2}}{3}\right)^3 \text{ cubic inches,}$$

and 1 hexahedron occupies—

$$\frac{(8)^3 \times 2\sqrt{2}}{3} = 6.6987 \text{ cubic inches.}$$

The specific gravities of these allotropic modifications should be inversely according to their space occupation. Thus, the regular form should have a specific gravity—

$$\frac{2.34}{1} \times \frac{5.6568}{6.6987} = 1.976.$$

The mean of the observed specific gravities is—

$$\frac{1826 + 2089}{2} = 1.957.$$

The difference between the estimated and mean results thus is less than the variations of observation.

Phosphorus also occurs in a very stable amorphous form, and, as such, might be accounted for by hexahedra face to face, so that two zonal and two apical solid angles

are in apposition. In such a grouping, continuous form is impossible. Its sp. gr. varies from 1.954 to 2.293 at ordinary temperatures.

The rhombohedral form, or that form in which the different sort of solid angles are in contact, is the most stable form, and one acting chemically with difficulty. The regular form very easily enters into chemical action. That the rhombohedral form is the most stable is a most significant fact.

The valence of phosphorus toward oxygen, sulphur, and chlorine is five; toward hydrogen, three. All the areas of valence are available to oxygen, and but three to hydrogen. Hydrogen is an electro-positive element, and the area to which it is attached must be electro-negative in sort. The two remaining angles or areas of valence do not attract hydrogen, and so are of similar electro-condition. Thus there are angles three of a sort and two of a sort in form, and three of a sort and two of a sort in valence or electro-condition; and an arrangement in which the three of a sort were in junction with the two of a sort in form, is more stable than an arrangement in which the three of a sort are grouped together, and the two of a sort in form together. Is it not a fair inference that the three of a sort in form are the three of a sort in valence or electro-condition, and, similarly, the two of a sort in form the two of a sort in electro-condition, and that the zonal angles are electro-negative and the apical electro-positive, and that the angle nearer the centre is electro-negative to the more distant one?

Arsenic, antimony, and bismuth occur in rhombohedra with an increasing angle R. Arsenic and antimony occur rarely in the regular form.

Sulphur has a valence of six, and would thus have a primal form of six solid angles. Of such a form are octahedra. If octahedra be placed with their similar edges together, so that if there be any difference of solid angles different sorts are in apposition, they will reproduce themselves in gross form. Sulphur occurs in rhombic octahedra of sp. gr. 2.05, and axes 1, 1.23, 2.344, and so we may assume the atomic form to be of these proportions. Sulphur also occurs in a form said to be monoclinic, but really in a clinical form.

This modification occurs at higher temperatures than the rhombic one, is of sp. gr. 1.96, and at ordinary temperatures reverts with the evolution of heat to the rhombic form. It occurs in brilliant, yellow, transparent, columnar crystals of oblique, basal cleavage. Thus the clinical form of sulphur is in a condition of strain, has a slight lowering of specific gravity, has interatomic spaces through which the atoms rotate to assume the rhombic arrangement, and the primal form must account for these properties in such a way that the specific gravities are inversely to the space occupied per form. These primal forms may be placed face to face, with different zonal and solid angles in apposition, as in the rhombic arrangement. In this case the apical solid angles diverge, and make continued form impossible. If these become convergent, the zonal angles are slightly displaced, and so in strain; the zonal edge of the atom form becomes less oblique to the lines of fission into series, thus thickening the series 1/20.3, approximately the observed proportionate decrease of specific gravity. In such an arrangement there are spaces permitting the forms to diverge, until their long axes become parallel to assume the rhombic arrangement. The arrangement face to face, as described, gives a clinical columnar form of oblique basal cleavage.

Selenium has modifications similar to those of sulphur. Tellurium occurs in rhombohedra, and might be accounted for by regular octahedra placed face to face. This view is supported by its isomorphism with the regular elements, gold and silver. The hydrides of these elements will be discussed in connection with that of chlorine.

In sulphur the number of atoms necessary to give a complete rhombic and clinical group, is the number of atoms found in their respective gas molecules. The six-atom group occurs at a lower temperature than the

two-atom group, in both crystalline and gaseous conditions.

Iodine crystallises from the molten state in foliated masses, tenacious in two planes, and easily fissile in the third, to give plates showing striæ intersecting at angles of 70° . Thus is shown a slight attraction in one axis, and a very much stronger attraction in the other, or those in which the series are at 72° to each other.

Iodine may be seen under the microscope to crystallise (from its solutions in diethyl ether and chloroform) in rhombic octahedra and platelets, in needles, and in roughly rhombic retiform combinations. The octahedra occur in slowly evaporating concentrated solutions, and their like edges meet over the most acute solid angle, at 72° and 54° respectively. The platelets occur in more rapidly evaporated solutions, and in company with octahedra or needles, according to the slowness of the evaporation. The platelets have angles 72° and 108° , and the acute angles of one platelet when it comes in the vicinity of another platelet shows an attraction and choice for the obtuse angles of the other platelet, and *vice versa*.

The needles occur in the more rapidly evaporating solutions. If in a solution two needles, from opposite directions, project themselves toward the same point, as they approach they cease their self-projection, and thicken themselves while there remains iodine in solution. Should, however, a needle project itself toward the more central portion of another needle, it will continue to grow towards and will join the other needle at an angle 72° or 36° . The needle may even take a curved course to arrive at that angle, and will remain curved until the solvent is evaporated, after which it assumes a straight form. While the crystal is taking the iodine from the solution, the colour is seen to go at first slowly, and decrease with accelerated velocity, until it disappears almost instantaneously, while there yet remains present some of the solvent. The rhombic retiform body is cast down on still more rapid evaporation, and consists of bars of iodine which appear fine under 450 D enlargement. If this form be permitted to evaporate, as the bars which connect some outer bar volatilise, and the outer bar is not held off by intervening bars, the outer bar springs with great velocity to the inner portions of the retiform body, showing an attraction at sensible distance.

Iodine has a valence of seven, and so its atom form may be accounted for by a bipyramidal dekahedron,—that is, a form of seven solid angles. Its symmetric form would be of five zonal angles at equal distances apart, and two apical angles, one upon either side of and equidistant from the zone, and each apical solid angle equidistant from the zonal solid angles. If such a form be placed with a zonal edge in apposition with the zonal edge of another form, and their zonal planes in the same plane, a rhombic group of angles 108° and 72° in the zonal plane results. These groups may be so arranged that they give a gross form of these proportions. If such a layer of atoms be placed over a layer, but with one series less than the layer beneath, so that the apical angles of each layer are in contact, and layer be placed over layer with a similar decrease of series, the half of an octahedron will be formed. If the primal form have its apical solid angles the same distance apart as the zonal angles, and the other half of the octahedron as above described be built, an octahedron, in which similar edges over the most acute solid angle will make 72° and 5 yards of an angle, will result. Such an arrangement permits of union of single attractive areas in the one axis, and five attractive areas in the other two axes, thus accounting for the easy fission into tenacious layers of angles 72° and 108° .

The hydrides of this group are monohydrides. There are compounds of the formulæ Ag_2Cl and K_2Cl , showing a bivalence toward elements of Group I. of Mendeleeff's Table, and two particularly electro-negative areas of valence, or of the series which corresponds in number with those, which may be shown in iodine to be nearest

the centre of the atom. The satisfaction of one of these areas of valence satisfies the opposite one. If such a condition holds true in sulphur, then the hydride of sulphur would be the observed one, sulphur dihydride.

The elements of Group VIII. have a valence of four, and so would, if the primal form be a regular tetrahedron, give rise to regular forms. Such crystal forms as have been observed are of regular sort. There may be shown certain reasons to believe that both oxygen and sulphur may have a monovalent action, and that in osmium and iridium tetroxides the oxygen is monovalent.

The remaining Groups of Mendeleeff's Tables, III., II., and I., would have as primal forms a triangle of thickness, a rod form, and a spherical form, respectively. The triangular form in aluminium may be shown to be equilateral, in that two atoms are attached by three atoms of oxygen, and these outlying points formed by the oxygen atoms, when grouped, give a hexagonal form, which must be dependent on equilateral triangular subforms, the grouping of which is dependent on the central or aluminium atoms, which dictate the arrangement. These triangles may lie in the planes of the faces of the tetrahedra when arranged in the diamond grouping.

The atoms of the elements of Group II. add themselves up to give, first, equilateral triangles; and second, these triangles to give either regular or hexagonal groups. Both regular and hexagonal forms occur in zinc, and the hexagonal in beryllium. Cadmium and mercury occur in the regular system.

The atoms of Group I. may unite, first, in rods of two atoms; second, in a rod of two rods of the first sort; third, three rods to form an equilateral triangular body; and fourth, these triangles together to give a regular form. These four steps are curiously coincident in number with the four allotropic modifications of silver, the first being smallest and easiest of solution, the second more difficult of solution, the third more stable in form, and the fourth the most stable of arrangements.

Malleability has a coincidence with atomic forms, permitting of interatomic mobility in their arrangement. Thus the atoms of carbon are of regular tetrahedral form, and any loose solid angle is as an apex to a tripod, of equal limbs. Hence the stability of form and the rigidity of the diamond. If one angle of an aluminium atom become free, it may, unless checked by some other atom, rotate circularly about a line joining the two remaining angles. Thus aluminium shows a marked interatomic mobility, by its malleability, and its tendency to a variable crystal form. If, in an atom of an element of Group II. of Mendeleeff's Table, one of its two areas of attraction be free, it may, unless checked by some other atom, rotate spherically about its stationary area of attraction. The interatomic mobility of such elements is shown by their increase of malleability over the elements of Group III. The interatomic mobility and malleability of the elements of Group I. are increased over those of Group II. by a joint in their rod form.

There is another factor in interatomic mobility—the intensity of attraction. If chemical and crystal attractions be different manifestations of the same attraction, then with the decrease of chemical activity there will be a decrease of rigidity and stability of form, with an increase in interatomic mobility and malleability.

The preceding relations are well shown in the following comparisons:—

- { The diamond.—Very crystalline, brittle, and hard.
- { Tin.—Malleable, breaking with crystalline structure.
- { Lead.—Soft and malleable.
- { Iron, nickel, and cobalt.—Brittle as compared with platinum.
- { Magnesium, zinc, cadmium, and mercury.—Increasing in softness, with increase of atomic weight and decrease of chemical activity.
- { Copper, silver, and gold.—Increasing in malleability with increasing atomic weight and decreasing chemical activity.

Thus is given a set of relations evidencing the unity of chemical and crystal attractions.

It is probable that, at no distant day, we will be able to determine the causes of the different physical properties of different sorts of matter, and that, in the determination of these causes, form will bear no insignificant part.

In closing this very incomplete sketch it might be well to add that, within a short time, the original paper will be published in full.

Byng Inlet, Ontario, Canada.

ON THE SEPARATION OF THE OXIDES OF CERIUM AND THORIUM.*

By G. WYROUBOFF and A. VERNEUIL.

(Concluded from p. 246).

Estimation of Cerium in the presence of other Rare Earths.—The methods we have described enable us to separate cerium quantitatively, with an accuracy superior to that of all previously known methods. It has the further advantage of not requiring much of the substance to be analysed.

Estimation in presence of Lanthanum, Didymium, and the Rare Earths of Yttria.—If the mixture of the oxides from the calcination of the oxalates was entirely, or only partially, soluble in nitric acid (which shows that it contains more than 50 per cent of cerium), it becomes easily soluble by the addition of small successive quantities of peroxide of hydrogen. A disengagement of oxygen, and the reduction of the cerosceric oxide, takes place. To the more or less violet limpid solution we add peroxide of hydrogen (10 c.c. per grm. of the oxide), then ammonia until alkaline, and boil to transform the brown precipitate into the clear yellow cerosceric hydrate. This transformation is complete when there is no longer any disengagement of oxygen. The solution is then filtered and washed, to get rid of the nitrate of ammonia which is formed by the last reaction. Dry at 110°, and detach the filtrate from the paper as carefully as possible; the filter must be incinerated and calcined, and the residue treated with a few drops of HNO₃ hot, and peroxide of hydrogen, and then evaporated to dryness. It is taken up with a little water and added to the solution of the bulk of the precipitate, which must be dissolved in warm nitric acid. This solution is evaporated down to a syrup, taken up with a few c.c. of water, and again evaporated to dryness. On cooling, a vitreous mass of a deep yellow colour is obtained; this is dissolved in 100 c.c. of a 5 per cent solution of NO₃NH₄, and heated to about 70°; the yellow liquid begins to get clouded, a yellowish white precipitate forms, and the reaction is finished when the supernatant liquor takes the violet tint of the didymium salts. After filtering, the precipitate is washed with a 5 per cent solution of NO₃NH₄ and calcined. We thus obtain 75 or 85 per cent of the cerium present in the mixture, in a perfectly pure state. The filtrate is precipitated by oxalate of ammonia, of which much excess should not be used, the oxalates of Ce, La, and Di, contrary to the general opinion, being very slightly soluble. The solution is made alkaline by a few drops of ammonia. The oxalates, which do not need to be washed, are calcined; this time they are all soluble in HNO₃. On repeating the above operation one can obtain several hundredths of pure cerium. The filtrate contains the rest of the cerium and all the other earths; these are re-converted into oxalates and calcined. The substance is then dissolved in HNO₃, a few drops of peroxide of hydrogen added to reduce the cerium, and then evaporated to dryness. To the solution of the nitrates we add acetate of soda, and precipitate by

a large excess of peroxide of hydrogen (about 10 c.c. of water). The liquid must be slightly warmed to hasten the deposition of the precipitate, which contains all the cerium as well as a small quantity of the other earths. It is then washed with water to which a few drops of peroxide of hydrogen have been added. The filtrate is then precipitated either by oxalate of ammonia or by ammonia. The following figures show the degree of exactness possible by this process:—We took 1.242 grms. of a synthetic mixture, containing 0.3718 grm. of Ce₂O₃ and 0.8702 grm. of other earths, such as are found in monazite after the elimination of the thorina and cerium.

1st precipitation ..	0.3160	pure Ce ₂ O ₃ .
2nd " ..	0.0175	
3rd " ..	0.0485	Ce ₂ O ₃ + LaO, DiO.
	0.3820	

Thus we recovered the cerium with a surcharge of 2.68 per cent, due to the entanglement of the other earths in the precipitation by peroxide of hydrogen in acetic solution.

Estimation in the presence of Thorina.—It is necessary for the mixture to be in the state of nitrate. We therefore dissolve the oxalates in nitric acid; although the pure oxalate of thorium is insoluble, the solution is easily made when the mixture contains 50 per cent of cerium. If the proportion of thorium present is greater, the oxalates must be decomposed by soda; in the presence of ThO, oxalate of cerium, which is attacked with difficulty when by itself, is easily decomposed in the cold. The solution of the nitrates, which should not contain much more than 0.5 of oxides, especially if thorina predominates, is evaporated to dryness or neutralised with ammonia; the residue is dissolved in water, and 10 c.c. of distilled peroxide of hydrogen (at 10 volumes) is added; it is then heated for a few moments to about 60°. The very voluminous precipitate is collected on a filter and washed, until the wash-waters are no longer precipitated by ammonia. It contains practically all the thorina, and the filtrate contains all the cerium within a few hundredths. It is therefore necessary to treat both fractions a second time. For this purpose the cerium is precipitated with ammonia; the precipitate must be dissolved on the filter by means of a few c.c. of dilute HNO₃, which are run through several times; the acid should be first warmed. The solution is evaporated to dryness, and, as before, treated with peroxide of hydrogen. The small quantity of thorina thus obtained is added to the first precipitate; the cerium remaining in solution this time is perfectly pure; it can now be precipitated by ammonia, calcined, and weighed in the state of Ce₂O₃. The second treatment of the peroxide of thorium is much more simple. The precipitate cannot be dissolved on the filter, as the disengagement of oxygen would cause a slight loss owing to the tiny drops of liquid which would be thrown out by the bubbles of gas. It must be removed from the filter, as carefully as possible, by means of a glass rod—this is not a difficult operation, thanks to the gelatinous nature of the precipitate; it is then dissolved in 2 c.c. of HCl, to which 2 c.c. of water and 1 grm. of HNO₃ have been added; the reduction is instantaneous. The solution is then passed through the filter to dissolve any of the material which may still adhere to it. The filter is then washed quickly, and the solution precipitated with ammonia. The hydroxide is collected on the same filter, dissolved in HNO₃, and the solution, neutralised with ammonia, is precipitated with peroxide of hydrogen. The same filters will easily serve for all the operations. The one for the oxide of cerium, the other for the thorina.

The peroxide of thorium, which has been twice precipitated by peroxide of hydrogen, contains only insignificant quantities of cerium (about 0.1 per cent); this is quite sufficient for ordinary analyses. Unfortunately the Th₄O₇N₂O₅ collected cannot be calcined; it decrepitates

* From the *Bull. Soc. Chim.*, Series 3, vol. xix.—xx., No. 6.

very strongly at a high temperature, being transformed into an impalpable powder which cannot be kept in the crucible; this causes a loss of at least 10 per cent. It is therefore necessary to once more reduce it, by a mixture of HCl and INH_4 , precipitate by ammonia, and calcine to the state of hydroxide. All these operations, which appear to be very lengthy, do not really take up much time, thanks to the fact that most of the precipitates do not need any washing, for the reason that no matter is introduced which cannot be eliminated by calcination. We have given a few examples, showing the satisfactory nature of the results obtained by this method:—

	Taken.	Found.
1. ThO	0.3670	0.3655
Ce_2O_3	0.0221	0.0229
2. ThO	0.3645	0.3640
Ce_2O_3	0.0214	
LaO + DiO ..	0.0140	0.0354 0.0370

In the two following analyses the cerium was only precipitated once, and therefore contained a little thorium:—

	Taken.	Found.
3. ThO	0.0508	0.0475
Ce_2O_3	0.9485	0.9515
4. ThO	0.3677	0.3651
Ce_2O_3	0.0207	0.0238

Mr. Dennis (*Zeit. Anal. Chem.*, xiii., p. 412, 1897) has proposed a very interesting reaction for the separation of thorium and cerium. He precipitates the neutral solution of the nitrates by the potassic salt of hydronitric acid, and then boils for a few minutes.

The thorina is thus precipitated integrally in the form of hydroxide, which can be immediately calcined. The only inconvenience of this process, which is as simple as it is charming, is the total precipitation of the thorina, as this same thorina will carry with it some small quantity of cerium which cannot be removed by subsequent precipitations. It is easy to make certain of this by treating one of the solutions used in the manufacture of Auer mantles, by KN_3 ; they contain from 1 to 1.5 per cent of cerium. The whole is precipitated by this means, and the filtrate does not contain any cerium. For the purpose of comparing the two methods, we took a synthetic mixture containing 0.0952 of ThO and 0.0927 of CeO.

After precipitation by KN_3 , we found in the filtered solution 0.0896 of CeO quite free from thorium.

The thorina was re-dissolved in HNO_3 , and precipitated by peroxide of hydrogen; the filtered solution then gave 0.0028 of CeO.

The thorina thus carried with it 3.34 per cent of cerium which was recovered by means of peroxide of hydrogen.

The method we propose is therefore much more accurate than that suggested by Mr. Dennis.

THE ESTIMATION OF MANGANESE AS THE SULPHATE AND AS THE OXIDE.*

By F. A. GOOCH and MARTHA AUSTIN.

THE estimation of manganese by the conversion of salts of that element with volatile acids to the form of the anhydrous sulphate by the action of an excess of sulphuric acid, evaporation, and gentle heating, was formerly a recognised procedure. On the authority of Rose (*Ann. Phys. Chem.*, cx., 125), however, this method was set aside on account of the supposed difficulty of removing the excess of acid without disturbing the composition of the normal salt. Thus, Oesten, working under Rose's direction, obtained, upon submitting the crystalline

hydrous sulphate $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$ to heat, results which may be summarised and compared with the results obtained by Rose's sulphide method (the ignition of the residue with sulphur in hydrogen) as given in Table I.

The residues remaining after the gentle ignition of the sulphate weighed apparently several m.grms. more than should have been the case if the salt had been reduced to the normal anhydrous sulphate. At higher temperatures the sulphate turned brown and lost altogether too much weight. A comparison of the errors of the process in which the ignition was at low temperature with those of the sulphide process would seem to justify Rose's rejection of the former method for the latter. Upon re-calculating these results, however, using atomic weight in use at present—viz., Mn=55, S=32.06, O=16—it becomes plain that the errors of the two processes, as shown in Oesten's work, are not very different numerically, though with opposite signs (Table II.).

The most uncertain element in these experiments is the difficulty, well-recognised at present, of getting the hydrous manganous sulphate, upon which the experiments were made, in a perfectly definite condition of hydration.

Volhard (*Ann. d. Chem.*, cxviii., 328) subsequently studied the sulphate process, and showed that manganous sulphate may be dehydrated, separated from an excess of sulphuric acid, and brought into definite condition for weighing as the anhydrous salt by careful and protracted heating over a special device of his own—a ring burner enclosed in a sheet-iron casing. Thus, on evaporating and dehydrating a solution of pure neutral manganous sulphate, Volhard obtained the results recorded in the following statement:—

Residue of MnSO_4 left by evaporation and dehydration	0.1635
Residue after treatment with three drops of H_2SO_4 and heating three hours ..	0.1635
Residue after heating two hours	0.1638
Residue after treatment with four drops of H_2SO_4 and heating two and a half hours	0.1635
Residue after heating three hours	0.1635

Similar results were obtained on evaporating with sulphuric acid and igniting in like manner an aqueous solution of manganous chloride. Volhard's recommendation of the method has not secured for it the acceptance which its simplicity and exactness would seem to demand—possibly because the periods of ignition appear to be considerable, and the manner of heating special.

In our own experiments with the sulphate process we have found that special apparatus is unnecessary, that the time of treatment may be short, and that the process is in every respect simple as well as very exact. We took for a starting-point manganous chloride prepared in the manner to be detailed. An aqueous solution of the so-called pure manganous chloride of commerce was boiled with pure manganous carbonate (to throw out aluminum, iron, and chromium), filtered, and precipitated with ammonium sulphide. The precipitate thus obtained was dissolved in a very slight excess of hydrochloric acid (to leave behind possible traces of nickel, cobalt, and copper), the solution was boiled to expel hydrogen sulphide, and precipitated with sodium carbonate. The manganous carbonate thus thrown down was boiled repeatedly with successive portions of water, and washed until the washings were free from chloride. The greater part of this purified carbonate was dissolved in the least possible amount of pure hydrochloric acid, the reserved portion of the carbonate was added, the mixture was boiled, and the solution of the purified and neutral manganous chloride was filtered from the excess of undissolved carbonate. Definite portions of this solution were precipitated with silver nitrate, and from the weight of the silver chloride thus obtained the amount of manganous chloride present was calculated. Portions of the solution thus standardised

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, vol. v., p. 209.

TABLE I.

MnSO ₄ ·5H ₂ O taken. Grm.	MnSO ₄ found. Grm.	Theory. Grm.	Error. Grm.	MnS found. Grm.	Theory. Grm.	Error. Grm.
1·659	1·043 (a) 1·023 (b)	1·087	0·008 + 0·014 -	0·597	0·595	0·002 +
1·481	0·934 (a) 0·905 (b) 0·725 (c)	0·928	0·008 + 0·021 - 0·201 -			
1·430	0·880 (b)	0·893	0·013 -	0·509	0·512	0·003 -
(a) Ignited gently.		(b) Ignited at low red heat.		(c) Ignited at strong red heat.		

TABLE II.

MnSO ₄ ·5H ₂ O taken. Grm.	MnSO ₄ found. Grm.	Theory. Grm.	Error. Grm.	MnS found. Grm.	Theory. Grm.	Error. Grm.
1·659	1·043	1·039	0·004 +	0·597	0·599	0·002 -
1·481	0·934	0·928	0·006 +			
1·430				0·509	0·516	0·007 -

TABLE III.

MnSO ₄ calculated from AgCl found in 50 c.m. ³ of solution A. Grm.	MnSO ₄ found by treatment of 50 c.m. ³ of solution A with H ₂ SO ₄ . Grm.	MnSO ₄ found by treatment of 50 c.m. ³ of various solutions with H ₂ SO ₄ .					
A.	A.	B.	C.	D.	E.	F.	G.
1. 0·3518	1. 0·3513	1. 0·3100	1. 0·3256	1. 0·3534	1. 0·3524	1. 0·3355	1. 0·5475
2. 0·3512	2. 0·3514	2. 0·3104	2. 0·3254	2. 0·3543	2. 0·3520	2. 0·3357	2. 0·5476
	3. 0·3518	3. 0·3096					

were drawn, for our experiments, from a burette into a weighed platinum crucible, sulphuric acid was added in amount more than equivalent to the manganese, the solution was evaporated on the water-bath until the water was removed, and then, supported by means of a porcelain ring or triangle, within a larger porcelain crucible used as a radiator, so that the bottom and walls of one were distant from the bottom and walls of the other by an interval of about 1 c.m., the crucible was heated more strongly. The outer porcelain crucible may be heated over a good Bunsen flame to a red heat without risk of over-heating the manganese sulphate within the inner crucible, and the ignition may proceed as rapidly as is consistent with the avoidance of mechanical loss by spattering. The results obtained by treatment of equal portions (50 c.m.³) of the same solution are given, together with the results of standardising the solution by precipitation with silver nitrate, in columns A (Table III.). In the other columns are given comparative results got in the treatment of equal portions of several other solutions employed subsequently in other work.

These results show plainly that the process of estimating manganese in the form of the anhydrous sulphate is both simple and accurate.

The estimation of manganese as the manganoso-manganic oxide, Mn₂O₄, has been so frequently criticised unfavourably that the method may be said to have passed from very general use, excepting in certain cases in which the directness of the process is a temptation to incur the risk of some uncertainty. The production of the other oxides of manganese in definite condition is thought to be even more uncertain. Manganese dioxide, MnO₂, begins, as Wright and Menke have shown (*Journ. Chem. Soc.*, xxx., 775) to lose oxygen at a temperature (about 210° C.) to which the hydrated oxide must be heated to free it from water, or very nearly that at which the nitrate is converted into the dioxide; so that the chance of producing an undecomposed dioxide by the ignition of the hydrated dioxide (the form in which the dioxide generally appears in analytical processes), or of the nitrate, is small. Manganic oxide, Mn₂O₃, is produced, it is said, from the other oxides by ignition at a low red heat under the ordinary conditions. The manganoso-manganic oxide, Mn₂O₄, forms, presumably, when an

oxide of manganese is submitted, under ordinary atmospheric conditions, to the high heat of the blast-lamp. If the proportion of oxygen in the surrounding atmosphere is reduced below the normal, the conversion of Mn₂O₃ to Mn₂O₄ goes on very easily, as Dittmar has shown (*Journ. Chem. Soc.*, xvii., 294), at a temperature between the melting points of silver and aluminum, while if the proportion of oxygen in the surrounding atmosphere falls much below the normal, the reverse change, from Mn₂O₄ to Mn₂O₃, tends to take place at the same temperature. It is not surprising, in view of these phenomena, that the estimation of manganese as the oxide Mn₂O₄ should have fallen into disrepute; and yet, if the condition most favourable to the production of that oxide—a low proportion of oxygen in the surrounding air—can be maintained during the ignition, it is not impossible that the indications of the process might prove to be, under the conditions, reasonably accurate. Now, this may be exactly the condition of affairs when the ignition takes place ordinarily; for if the products of combustion displace the ordinary air about the crucible, the proportion of oxygen about the oxide falls to a low limit. We have made the experiment of enclosing the ignited crucible within an inverted crucible, so that the products of combustion should be held immediately about and above the ignited oxide, but our experience has shown that the object in view is attained, apparently, quite as well when the ignition is so arranged that the crucible simply rests well within the upper part of the flame of a strong Bunsen burner, or blast-lamp, in such manner that an oxidising flame covers nearly the entire wall of the crucible.

In the following experiments we have put to the test this matter of getting definitely the different oxides of manganese. We started with a known amount of pure anhydrous sulphate, prepared from the pure chloride in the manner previously described. This sulphate was converted by ignition into the oxide—presumably the oxide Mn₂O₄—the containing crucible being well within the upper flame of a powerful burner.

In the next step, this oxide was further oxidised by moistening it with nitric acid and heating the residue gently until the evolution of fumes ceased, the containing crucible being placed well above a porcelain crucible used as a radiator and heated so that only the bottom showed

[illegible]

a faint red heat. In this process the attempt was made to arrest the ignition at the point where the anhydrous dioxide was produced. As the table shows, and as would be expected, this attempt was only occasionally and partly successful.

The residue of the last process was then submitted to a higher heat. The platinum crucible containing the oxide was placed within and touching the bottom of a larger porcelain crucible which was heated to redness. Under these conditions the temperature should not be too hot, and the products of combustion should naturally be thrown so far away from the oxide undergoing ignition that circumstances should be favourable for the formation of the oxide Mn_2O_3 . The event proved that the attainment of the exact condition corresponding to the symbol Mn_2O_3 is a matter of some uncertainty.

Next, the oxide was subjected to the highest heat of a strong Bunsen burner (or in some cases, the broad flame of a blast lamp), the crucible being well surrounded by the products of combustion. The results of this treatment, it will be seen, agree, with a single exception out of ten experiments, reasonably well with the theory for Mn_2O_4 . By treating the final oxide with nitric acid and repeating the cycle of operations described, the observations of the phenomena were multiplied, until finally, the oxide formed last was treated with sulphuric acid, ignited in the manner previously detailed, and weighed as the anhydrous sulphate, thus showing that no significant loss of material had taken place in the series of manipulations. Table IV. comprises the results of these experiments. The Roman numerals indicate the order of treatment.

The inference is plain that the estimation of the manganese in the form of the manganoso-manganic oxide, Mn_2O_4 , is by no means to be considered utterly untrustworthy when the process is conducted in the manner described, though it must be recognised that an irregular result may occur occasionally. The danger of accepting such an irregularity as a correct indication may be eliminated to a very considerable extent if the precaution is taken invariably to moisten the ignited oxide with nitric acid, and ignite again. The indications of harmonious results thus got may be taken with a fair degree of confidence. However, it is, in our judgment, by far the wiser and simpler plan to convert an oxide of manganese obtained in course of analysis into the sulphate and to weigh the manganese in that form.

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, May 19th, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

MESSRS. T. M. Lowry, H. St. John, and J. Heaton were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of
favour of Messrs. John Brighouse Buchan, 17, Steven
Street, Stretford, Manchester; Charles William Tisdale
Davies, Wye, Kent; Robert Findlay Hialop, Craigieola,
Paisley; George Herbert Martin, New College, East-
bourne; Charles James Meads, Brooklyn, Erlanger Road,
St. Catherine's Park, S.E.; Matthew Joseph Sheridan,
20, Heathland Road, Stoke Newington, N.

The following papers were read : —

79. "*The Liquefaction of Hydrogen and Helium.*" By JAMES DEWAR, LL.D., F.R.S.

In a paper entitled "The Liquefaction of Air and Research at Low Temperatures" (*Proc.*, 1895, xi., 221), an account was given of the history of the hydrogen

problem and the result of the author's experiments to the end of the year 1895. The subject was again discussed in a Lecture on "New Researches on Liquid Air" (*Proc. Roy. Inst.*, 1896, xv., i., 144), and a sketch given of the apparatus employed for the production of a jet of hydrogen containing liquid. It was shown that such a jet could be used to cool substances below the temperature which could be reached by the use of liquid air, but all attempts to collect the liquid in vacuum vessels failed. The type of apparatus used in these experiments worked well, so it was resolved to construct a much larger liquid air plant, and to combine with it circuits and arrangements for the liquefaction of hydrogen. This apparatus, admirably constructed by the engineers, Messrs. Lennox, Reynolds, and Fyfe, took a year to build up, and many months were occupied in testing and making preliminary trials. The many failures and defeats need not be detailed.

On May 10th hydrogen was liquefied by allowing the gas, cooled to -205° , and under a pressure of 180 atmospheres, to escape continuously at the rate of from 10 to 15 cubic feet per minute from the nozzle of a coil of pipe in a double silvered vacuum vessel of special construction surrounded with a space kept below -200° . Liquid hydrogen commenced to drop from this vacuum vessel into another doubly isolated by being surrounded with a third. On this occasion 20 c.c. of liquid hydrogen were collected in about five minutes, and on May 12th 50 c.c. were obtained before the hydrogen jet froze up from the solidification of air in the pipes. The yield of liquid was about 1 per cent of the gas. The hydrogen in the liquid condition is clear and colourless, showing no absorption spectrum, and the meniscus is as well defined as in the case of liquid air. The liquid must have a relatively high refractive index and dispersion, and the density appears to be in excess of the theoretical value, 0.18 to 0.12, deduced respectively from the atomic volume of organic compounds and from the limiting density found by Amagat for hydrogen gas under infinite compression. The author's experiments on the density of hydrogen in palladium gave a value 0.62 for the substance in combination, and it will be interesting to find the density of the actual liquid at its boiling-point. Not having arrangements at hand to determine the boiling-point, two experiments were made to prove the excessively low temperature of the boiling fluid. In the first place, a long piece of glass tubing, sealed at one end, but open to the air at the other, and cooled by immersing the closed end in liquid hydrogen, immediately filled, where it was cooled, with solid air. The second experiment was made with a tube containing helium.

The *Bulletin* of the Cracow Academy for 1896 contains a paper by Professor Olsewski, entitled "A Research on the Liquefaction of Helium," in which he states that "As far as my experiments go, helium remains a permanent gas, and apparently is much more difficult to liquefy than hydrogen." Helium which had been extracted from Bath gas by the liquefaction method described last year (*Proc.*, 1897, xiii., 190), and sealed up in a bulb with a narrow tube attached, was placed in liquid hydrogen, and a distinct liquid was seen to condense. A similar experiment made with the same helium tube in liquid air under exhaustion instead of in liquid hydrogen gave no visible condensation. It would thus appear, as already suggested by the author (*loc. cit.*), that there cannot be any great difference in the boiling-points of helium and hydrogen. A fuller account of the work will appear in the *Transactions*.

All known gases have now been condensed into liquids which can be manipulated at their boiling-points under atmospheric pressure in suitably arranged vacuum vessels. With hydrogen as a cooling agent, it will be possible to get within 20° or 30° of the zero of absolute temperature, and its use will open up an entirely new field of scientific inquiry.

During the whole course of the low temperature work

carried out at the Royal Institution, the invaluable aid of Mr. Robert Lennox has been at the author's disposal, and it is not too much to say that, but for his engineering skill, manipulative ability, and loyal perseverance, the present successful issue might have been indefinitely delayed. The author's thanks are also due to Mr. J. W. Heath for valuable assistance in the conduct of these experiments.

DISCUSSION.

Sir WILLIAM CROOKES proposed that the Fellows of the Society should offer their best congratulations to the President on the striking success which had attended his remarkable attempt to liquefy the last of the "permanent" gases—hydrogen and helium.

Dr. ARMSTRONG, in seconding the proposal, said that in the future the fact that the liquefaction of hydrogen had been achieved by a President of the Society would no doubt be recalled with interest. In early days, the discussion of the properties of hydrogen had attracted much attention in the Society, when Sir Benjamin Brodie advocated the view that it must be supposed to have metallic properties, a view subsequently strongly supported by Graham's discovery of hydrogenised palladium. He ventured to think, however, that the subject had been regarded too much from the inorganic side, and that when the evidence to be derived from organic chemistry was taken into account it was more probable that hydrogen would be found to resemble the petroleum hydrocarbons rather than the metals.

He then asked whether the President's statement that argon solidified when cooled in liquid air did not preclude the presence of argon in the helium from Bath gas, which gave only a liquid when cooled in hydrogen.

The motion was carried by acclamation.

The PRESIDENT, in acknowledging the compliment, said that the facts referred to by Dr. Armstrong proved that the helium employed in the liquefaction experiments did not contain argon. It was possible that hydrogen might be present in small quantity, but otherwise the helium was pure.

80. "The Action of Formaldehyde on Amines of the Naphthalene Series." (Part I.). By GILBERT T. MORGAN, B.Sc.

By the interaction of methylal, acetone, and β -naphthylamine, Reed (*Z. Prakt. Chem.*, 1887, xxxv., 314) obtained naphthacridine, $C_{21}H_{13}N$ (m. p. 216°) and a substance melting at $202-203^{\circ}$, to which he gave the formula $C_{24}H_{20}N_2$.

The author finds that when formaldehyde (40 per cent solution) acts on β -naphthylamine in alcoholic solution in the presence of hydrochloric acid, four basic substances are produced. Two of these have the composition corresponding with the formula $C_{21}H_{13}N$; one crystallises in orange needles melting at $225-226^{\circ}$, and the other in straw-coloured needles melting at 216° . The latter was found to be identical with naphthacridine.

The third substance crystallises in colourless prisms often showing cruciform twinning, melts at 203° , and is identical with the compound of this melting-point described by Reed. Its composition is expressed by the formula $C_{23}H_{18}N_2$, and, as the author has obtained it by the action of formaldehyde alone on β -naphthylamine, it follows that acetone plays no part in the condensation.

The fourth compound is obtained only in small quantity, crystallises in silky needles having a faintly yellow tinge, melts at $186-187^{\circ}$, and has the formula $C_{22}H_{16}N_2$. Its hydrochloride, $C_{22}H_{16}N_2 \cdot HCl$, and nitrate, $C_{22}H_{16}N_2 \cdot HNO_3$, have been prepared, and the base can be regenerated from either salt by means of aqueous alkalis.

The orange-coloured base melting at $225-226^{\circ}$ stands in very close relationship to the isomeric naphthacridine (m. p. 216°), and is converted into it both easily and quantitatively. Its behaviour with hydrogen chloride is remarkable. When it is triturated with concentrated

hydrochloric acid, or submitted to the action of the dry gas, it forms a green hydrochloride, from which the orange base is obtained; but when dry hydrogen chloride is passed into its solution in benzene or in glacial acetic acid, a yellow hydrochloride is produced, which yields naphthacridine (m. p. 216°). The orange base is probably a labile form of naphthacridine. Methods for preparing each form in quantity are given in the paper, and the hydriodide, methiodide, and ethiodide of naphthacridine (m. p. 216°) are described.

The products of the interaction between formaldehyde and α -naphthylamine have been examined with results which will be communicated in a subsequent paper, and the investigation is being extended to derivatives of both α - and β -naphthylamine.

DISCUSSION.

Prof. DUNSTAN pointed out that commercial formalin usually contains acetone, so that, unless the author had satisfied himself of the purity of the formaldehyde, the fact that the interaction of the commercial solution and β -naphthylamine gave rise to the condensation product did not necessarily tell in favour of the view that acetone was not concerned in the action.

Mr. MORAN, in reply, said that the formaldehyde solution employed could have contained only a small amount of acetone. The base melting at 203° is readily reduced to naphthacridine by means of hydriodic acid and phosphorus, and this fact not only supports the formula adopted by the author, but also—taken in conjunction with the large yield of the base when prepared from β -naphthylamine and formaldehyde—makes it improbable that acetone takes any part in the condensation.

81. "On the Constitution of Oleic Acid and its Derivatives." (Part I.). By FRANK GEORGE EDMED, B.Sc.

Oleic and elaidic acids, when oxidised by weak alkaline permanganate, yield two dihydroxystearic acids, melting respectively at 134° and 99° (Saytzeff, *J. Russ. Chem. Soc.*, 1885, xvii., 417). The author finds that, while these acids are the chief products of the reaction, pelargonic, azelaic, and oxalic acids are also formed at the same time. These acids result from the continued action of the oxidising agent on the dihydroxystearic acids, and no evidence could be obtained of the formation of sebacic, suberic, and caprylic acids, which, with azelaic acid, have been stated to be the products of the further action of permanganate (Spiridonoff, *J. Russ. Chem. Soc.*, 1887, xix., 646). The dihydroxystearic acid obtained from elaidic acid is much more easily oxidised than its isomeride, a fact which is consistent with the stereoisomeric formula ascribed to elaidic acid. The formation of pelargonic and azelaic acids affords additional evidence in support of the formula $\text{CH}_3(\text{CH}_2)_7\text{CH}:\text{CH}(\text{CH}_2)_7\text{CO}_2\text{H}$, for both oleic and elaidic acids.

Oleic acid was also oxidised with a weak solution of chromic acid; the action was very slow, and a considerable quantity of azelaic acid, but only a trace of pelargonic acid, was obtained. No other products could be isolated, and the action appears to be similar to that of the permanganate.

The products resulting from the fusion of oleic and of elaidic acids with potash were quantitatively examined, and it was found in both cases that the yield of palmitic acid was approximately theoretical, but that the quantity of acetic acid was extremely small. Oxalic acid was also produced in the reaction, and the amount exceeded that of the acetic acid, but neither pelargonic nor azelaic acids could be detected, nor could any dihydroxystearic acid be recognised, although from Wagner's explanation of the reaction (*Ber.*, 1888, xxi., 3353) this might be expected to be an intermediate product.

DISCUSSION.

Mr. HEHNER was by no means convinced that the double linkage occurred between the central carbon atoms in the oleic acid formula. No doubt oleic acid on oxidation formed azelaic acid by preference, but as by other

means—for instance, by treatment with sulphuric acid or zinc chloride—lacones were very readily obtained, it seemed possible that oleic acid was a labile compound, and that the double linkage had not a definite position in the molecule. He was astonished at the excellent yield of dihydroxystearic acid obtained by the author by means of alkaline permanganate; in the most favourable circumstances he had been able to obtain only about 40 per cent of the theoretical yield, and this, he thought, was also the experience of other workers. The oxidation by means of chromic acid might have succeeded better if a solution of chromic anhydride in acetic acid had been employed instead of a mixture of bichromate and sulphuric acid. As to the deficiency in the yield of acetic acid in the potash fusion, might not some of the acetate have decomposed into methane?

The question arose whether oleic acid could be regarded as a single substance. The number of isomerides possible in the case of compounds containing 18 carbon atoms was enormous, and, while many were well known among the lower members of the series, it was remarkable that when a chain of about 10 carbon atoms was reached, all reference to isomerides disappeared from the text-books, solely, in his opinion, because means for their separation had not been devised. At the same time indications existed—such as slight differences in the optical behaviour of oils of a similar degree of unsaturation, and in the solidifying points of the fatty acids separated from them—which pointed towards isomerism. It was essential to elucidate this point before studying the constitution of the substance called oleic acid, especially as most of the work which had been done was antecedent to the recognition of the existence of less saturated acids, such as linolic and linolenic acids and their homologues, which were almost invariably present with acids of the oleic class.

Dr. CROSSLEY was surprised to learn that a nearly theoretical yield of dihydroxystearic acid was obtained by oxidising oleic acid with permanganate, as in similar experiments he had never succeeded in obtaining more than 40 per cent of the amount theoretically possible. Referring to Wagner's explanation of the production of palmitic from oleic acid by fusion with potash, he pointed out that it received support from many cases in which the alkali seems to act simultaneously as an oxidising and reducing agent, and suggested that the fusion of oleic acid with soda might lead to interesting results, as caustic soda apparently does not exercise a reducing action.

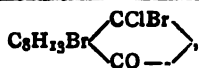
Prof. TILDEN said that, in his opinion, a body of evidence had been brought forward which amounted almost to positive proof of the correctness of the symmetrical formula for oleic acid. And, referring to the remarks of Mr. Hehner, he would venture to say that if it came to a question of the credibility of evidence, he should distinctly prefer the testimony derived from the action of a 1 or 2 per cent solution of permanganate in the cold to that of such agents as zinc chloride or caustic potash, which required a high temperature.

Prof. DUNSTAN doubted whether there was any real evidence that crystallisable oleic acid obtained from different oils is not a single substance. He thought it more probable that the differences Mr. Hehner had alluded to were due to impurities occurring in the natural oils.

Mr. EDMED, in reply, said that the quantitative yield of palmitic acid in the potash fusion precluded the supposition that the oleic acid employed contained a branched-chain isomeride. It was not possible to accept Mr. Hehner's suggestion that the small amount of acetic acid obtained was due to a decomposition of potassium acetate into methane during the reaction, as the temperature was not allowed to exceed 230° during the fusion.

82. "Stereoisomeric Derivatives of Camphor." By T. M. LOWRY, B.Sc.

In addition to the case of stereoisomerism referred to in a previous note (*Proc.*, 1897, xiii., 159), the author has investigated that of the dibromochlorocamphors,—



obtained by heating chlorocamphor with an excess of bromine in sealed tubes. When re-crystallised five times, the product melted at 81° and had a specific rotatory power $[\alpha]_D = 44.5^\circ$ in a 5 per cent solution in chloroform; after five more crystallisations, the rotatory power was raised to 51.3° , but no increase was observed after sixteen further crystallisations. The melting-point also was constant at 84° . There can be no doubt that the change in rotatory power indicates the presence of two stereoisomerides, and the product must, therefore, be an isomorphous mixture similar to that afforded by the bromochlorocamphors previously described, from which it differs only by the presence of an additional bromine atom in the β -position.

PHYSICAL SOCIETY.

Ordinary Meeting, May 27th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

A PAPER by Messrs. EDWIN EDSER and C. P. BUTLER on "*A Simple Method of Reducing Prismatic Spectra*," was read by Mr. Edser.

The production of interference-bands in a continuous spectrum is capable of furnishing a reference-spectrum which can be employed to determine the wave-lengths corresponding to the bright lines in a spectrum of a metal or of a gas. The authors discuss various methods by which such bands can be formed. In their final experiments, an air-film between two plane parallel glass plates is inserted in front of the slit of the spectrometer, in the path of the incident light. Owing to the interference of the direct ray with that twice internally reflected, bright bands separated by dark intervals are observed in the spectrum; these bright bands correspond to a series of different waves, whose lengths are easily determined for the whole series, when two of them are known. The bands are much improved by partial silvering of the two internal surfaces of the glass. It has been found that ordinary plate-glass, if well chosen, is good enough for all these experiments. In order to adjust for parallelism, a spot of light, or the filament of a glow-lamp, is viewed through the silvered surfaces. A long train of images is generally visible; these must be brought into coincidence. If now a sodium flame is looked at through the film, interference bands are seen. These bands must be adjusted by pressure, to be as broad as possible. An arc-lamp is used for illuminating the collimator slit. The authors exhibited the apparatus, and showed photographs of spectra-scales, with the appropriate wave-lengths calibrated upon them by this method. The results obtained were read from the spectrometer to 0.4 of a tenth-metre with an ordinary pocket lens. A simple graphic method enables wave-lengths, corresponding to a great number of spectral lines, easily to be determined by inspection. The phase-changes introduced by the silver do not affect the final result.

Prof. THRELFALL congratulated the authors on their discovery of a method that would greatly reduce the labour of calibrating spectra, and at the same time give such accurate results.

Prof. Boys said the simplicity of the apparatus added greatly to the value of the method. It would seem to him better if the slit were somehow contrived within the film-space. All want of definition due to rays falling at different angles upon the collimator object-glass would thus be avoided, and only a small part of the glass plates, i.e., the slit, would require to be strictly parallel planes. The limit of accuracy in the authors' method depended upon the collimator, not upon the optical perfection of the silvering of the plates.

Mr. BUTLER pointed out that previous methods had always required experienced spectroscopists for mapping out results. In the new method that work could easily be done by an assistant.

Mr. EDSER said that, by putting the two plates immediately in front of the slit, only a very small part of the glass is concerned in the action; light coming through at an angle would not reach the lens in the collimator.

Prof. Boys, Vice-President, then took the chair, and

Mr. CAMPBELL SWINTON read a paper on "*Some further Experiments on the Circulation of the Residual Gaseous Matter in Crookes Tubes*."

In the discussion that followed the former paper on this subject, at the Physical Society on March 25, 1898, Mr. Appleyard had suggested that, in tracing the cause of the rotation of the exploring mill, it would lead to simpler results if the vanes were made of some light conducting substance, for it was probable that mica introduced complications by retaining the charges. Prof. Boys then pointed out that the mica might be gilded. Such a tube has now been made by Mr. Wolff. With the gilded mica vanes so placed as to be outside the cathode stream, the mill behaves in a manner similar to the non-conducting insulated mill. It shows a greater tendency to assume a position of stability, due to electro-static induction; this renders it somewhat troublesome in starting, but when once under way, the mill rotates always when excited. Occasionally, when starting, a few reverse revolutions are observed. These are probably due to electro-static influence and momentum, and also possibly to eddy currents in the residual gaseous matter. But it is found, in all cases, that rotation in the direction that indicates a stream of residual gaseous matter from anode to cathode follows the reversal immediately after one or two oscillations. An electrometer connected to the mill through the pivot and needle-point, shows the vanes to be always electrified positively. The results are confirmed by a second tube with oblique vanes. The author concludes that at very high exhaustions there exists a molecular or atomic stream from anode to cathode, which carries a positive charge, and travels at high velocity outside the opposite cathode stream.

Mr. J. QUICK asked what was the minimum degree of exhaustion required to produce these results.

Prof. Boys said that the experiment gave some amount of probability to the truth of Mr. Campbell Swinton's hypothesis, but it did not altogether prove the mechanical theory of rotation to be correct. He was glad that a chance suggestion at the last discussion had led to such interesting experiments being continued.

Prof. THRELFALL mentioned that Boettger had devised a method for gilding mica, by a chemical process, that was much to be preferred to ordinary gilding.

Mr. CAMPBELL SWINTON said it was necessary to exhaust the tubes as completely as possible; to a point where it was only just possible for any discharge at all to pass through them. If the rotation was due to electrification, there must still be some mechanical process whereby the charges get to the vanes,—a stream of residual gas satisfied that condition.

The VICE-PRESIDENT proposed votes of thanks, and the meeting adjourned until June 10th.

Dinner of the Chemical Society.—In consequence of the death of Lord Playfair, the proposed dinner on June 9th, which was to be given by the Chemical Society to Lord Playfair and other Past Presidents who had been Fellows of the Society for fifty years, is postponed until the end of October. Dr. Mond's Garden Party on June 10th is also postponed.

DENSITY AND BOILING-POINT OF LIQUID HYDROGEN.

At the Chemical Society last night the President, Professor DEWAR, gave an account of his latest researches on liquid hydrogen.

Two of its physical constants have now been determined. Its boiling-point in air is -238°C ., or 35° absolute, determined by a platinum resistance thermometer; this value is higher by something like 17 per cent than the value obtained by Olzewski by adiabatic expansion. The whole range of liquid hydrogen is only about 50° from the absolute zero, because its critical point must be about 50° absolute. Thus it appears that there can only be a few degrees + or - in the different estimates based on theoretical conceptions as to its boiling-point. But these few degrees are really a large percentage of the whole, seeing that the range is so small. This value of the boiling-point enables us to predict with almost certainty that by no conceivable means at our present command shall we ever get nearer the absolute zero than -250°C ., or, say, $+20^{\circ}$ absolute; that is, the practical fall in temperature that liquid hydrogen will give under high exhaustion will never exceed 10° or 15° lower than its present boiling-point.

The next physical constant that has been determined is the density of hydrogen. As already described, the substance appears as a transparent fluid, with a well-defined meniscus, and it easily drops from one vessel to another, and may be said to collect with considerable rapidity. The appearance of the liquid would suggest a density probably not unlike liquid marsh gas, which has a density of 0.41 at its boiling-point in air, and is the least dense liquid known under such conditions. On allowing 10 c.c. of liquid hydrogen to evaporate, collecting the gas, and measuring its volume, the approximate density can be accurately determined. The resulting value comes to be a number rather less than 0.07. The result is, the liquid is about one-fourteenth the density of water, or it bears the same relation to water that hydrogen gas bears to air. The atomic volume, therefore, of liquid hydrogen is about 14; the atomic volume of liquid oxygen being about 13.7. The atomic volumes, therefore, of hydrogen and oxygen at their respective boiling-points are very close to each other. The density of the vapour of hydrogen at its boiling-point, assuming no abnormal condensation, is about half that of air, or approaches that of marsh gas at the ordinary temperature. The ratio of the hydrogen vapour at its boiling-point to the liquid is as 1 to 100, whereas in the case of oxygen the similar ratio is as 1 to 255.

Liquid hydrogen is in all respects the most extraordinary fluid chemists have ever had to deal with. No chemist could have anticipated that a liquid with a density of one-fourteenth that of water could have been capable of collection and manipulation in vacuum vessels with the same ease, practically, as manipulation with liquid air was carried on ten years ago, and that by its means we shall approach within 20° to 25° of the absolute zero.

On the Ammoniacal Bromides of Silver.—M. Jarry. —When submitted to the action of liquefied ammonia gas, dry bromide of silver loses its yellow colour and is transformed into a white powder; the excess of liquid is evaporated off, keeping the temperature at -30° . If now the temperature be gradually raised, at about $+4^{\circ}$ an abundant disengagement of ammonia is observed, though the powder still remains white; towards $+35^{\circ}$ another disengagement of ammonia gas takes place, and this time the bromide returns to its original yellow colour.—*Comptes Rendus*, cxxvi., No. 16.

OBITUARY.

LORD PLAYFAIR.

It is with great regret that we record the death of the Right Hon. Lord Playfair, K.C.B., F.R.S., which took place at his residence in Onslow Gardens on Sunday last, from bronchitis.

Lord Playfair has long been a prominent figure in scientific circles; he was one of the oldest Fellows of the Chemical Society, and would have been the principal guest at a dinner which had been arranged for the 9th of this month to entertain him and six other Past Presidents of the Chemical Society, all of whom have been Fellows for at least fifty years.

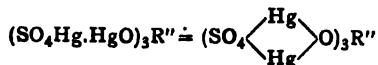
Lord Playfair was the son of Mr. George Playfair, Chief Inspector-General of Hospitals of Bengal, and was born at Meerut, Bengal, on May 21st, 1819; he was therefore just 79 years of age. After leaving St. Andrews, N.B., where he passed his school-days, he went to study chemistry at the Andersonian University, Glasgow, under Prof. Graham. In 1838 he went to Giessen, and there studied organic chemistry under Liebig, several of whose works he translated into English. In 1843 he went to Manchester, where he was appointed Professor of Chemistry in the Royal Institution; and in 1844, on the recommendation of the late Sir Robert Peel, he was appointed on the Commission constituted to examine into the sanitary condition of our large towns and populous districts, after which he was appointed Chemist to the Museum of Practical Geology. At the close of the Great Exhibition of 1851 he was made a Companion of the Bath, in recognition of his services as Special Commissioner in charge of the department of Juries, which post he again filled in 1862. In 1853 he was appointed joint Secretary, with Mr. Henry Cole, of the Department of Science and Art, and in 1856 he became Chief Inspector-General of Government Museums and Schools of Science. In 1857 Lord Playfair was elected President of the Chemical Society, and in 1858 he became Professor of Chemistry at the University of Edinburgh, where he had the honour of numbering the Prince of Wales and Prince Alfred among his pupils. He was one of the Royal Commissioners on the Cattle Plague, and was Chairman of the Royal Commission on the Scottish Fisheries. In 1874 he produced an elaborate scheme for the re-organisation of the Civil Service. He entered Parliament in the Liberal interest for the University of Edinburgh and St. Andrews in 1868, and in 1873-74 he held office as Postmaster General. In 1880 he was appointed Deputy Speaker of the House of Commons, and was made a K.C.B. on his retirement from this office and from the Chairmanship of Ways and Means in 1883. Lord Playfair was a Privy Councillor of the Queen, and of the Prince of Wales in the Duchy of Cornwall. He was a member of many learned societies and had many foreign orders; he was raised to the Peerage in 1892.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 16, April 18, 1898.

General Reaction of the Ethenic Carbides. Corresponding Mercuric Compounds.—G. Denigès.—By acting on butylene with mercuric sulphate in acid solution, compounds may be obtained corresponding to the general formula—



in which R'' represents an ethenic carbide. These compounds of a yellow colour dissolve easily in hydrochloric acid, accompanied by effervescence when the carbide combined with the mercury is volatile at the working temperatures.

Heat of Formation of some Quinones of High Molecular Weights.—A. Valeur.—The heat of formation of several quinones, starting from the corresponding hydrocarbides, are:—Ordinary quinone +129.1 cal., toluquinone +118.5 cal., thymoquinone +137.9 cal., α -naphthoquinone +138.1 cal., β -naphthoquinone +131.5 cal., anthraquinone +159.1 cal., phenanthraquinone +152.4 cal., retenequinone +167.2.

Heat of Neutralisation of Ethyl-phosphoric Acid.—G. Belugou.—The quantity of heat given off by the second molecule of alkali acting on the acid ethers is greater than that produced by phosphoric acid under the same conditions; it appears, therefore, that the di-potassic mono-phosphate ethers are less easily dissociable in aqueous solution than are the corresponding phosphates. Ethyl-phosphoric acid behaves in the same way as phosphoric acid towards helianthine and phthalein, and can therefore be estimated volumetrically.

Journal de Pharmacie et de Chimie,
Series 6, vol. vii., No. 6.

The Assay of Bronze used for Coinage.—A. Riche.—Reprint from the *Comptes Rendus*. Already noticed in this column.

The Simultaneous Volumetric Estimation of Sulphuric Acid and Lime in Waters.—L. Robin.—Already inserted in full.

The Detection of Rocou in Milk.—A. Leys.—The artificial colouration of milk is extending, especially by the addition of certain materials which impart to it a faint yellow tinge. If a sample of milk is suspected of containing *rocou*, 50 c.c. are treated in a flask with double its volume of a mixture of alcohol at 93°, 2400 c.c., ether 3200 c.c., water 200 c.c., and ammonia at 0.92 density, 80 c.c. After being thoroughly shaken, the flask is left quiet for a time, and the contents separate into two layers. The lower one, which consists of the casein and similar bodies in ammoniacal ethero-alcoholic solution, contains also the *rocou*, which imparts to the liquor a greenish-yellow tinge. After waiting for about twenty minutes, this lower layer is separated, and treated with half its volume of a 10 per cent solution of sulphate of sodium, which must be added in small portions at a time, while the flask is kept constantly shaken; a voluminous white precipitate, which quickly rises to the surface, is formed. By this means the greater part of the casein is removed without interfering with the colouring matter, which still remains in solution; this can then be extracted by means of amyl alcohol. After further treatment, a strip of cotton, which has been placed in the solution during evaporation, will, if *rocou* is present, immediately take a pink colour, while in the opposite case it will be yellow, after being dipped in a solution of citric acid.

MEETINGS FOR THE WEEK.

- MONDAY, 6th.—Society of Chemical Industry, 8. "Conditions existing in Acetylene Generators," by Prof. V. B. Lewes.
TUESDAY, 7th.—Royal Institution, 3. "Literary Criticism in Greece," by Professor S. H. Butcher, LL.D.
THURSDAY, 9th.—Royal Institution, 3. "Modern Methods and their Achievements in Bacteriology," by Edward B. Klein, M.D., F.R.S.
FRIDAY, 10th.—Royal Institution, 9. "Some Experiments with the Telephone," by The Right Hon. Lord Rayleigh, F.R.S., &c.
SATURDAY, 11th.—Royal Institution, 3. "The Temples and Ritual of Asklepios at Epidaurus and Athens," by Richard Caton, M.D., F.R.C.P.

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Further particulars may be obtained on application to the REGISTRAR.

PATENTS, DESIGNS, AND TRADE MARKS ACTS, 1883 TO 1888.

NOTICE IS HEREBY GIVEN, that

JAMES YATE JOHNSON, of 47, Lincoln's Inn Fields, London, has applied for leave to amend the Specification of the Letters Patent No. 18221 of 1896, for "Improvements in the manufacture and production of phthalic and sulpho-phthalic acids."

Particulars of the proposed amendments were set forth in the Illustrated Official Journal (Patents) issued on the 25th May, 1898.

Any person or persons may give notice of opposition to the Amendment (on Form G), at the Patent Office, 25, Southampton Buildings, London, W.C., within one calendar month from the date of the said Journal.

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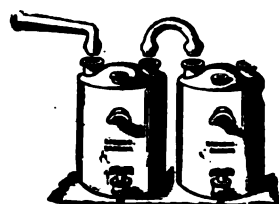
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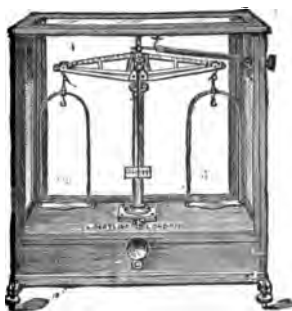
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THE CHEMICAL NEWS.

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ON THE LINDO-GLADDING METHOD OF DETERMINING POTASH.

By A. L. WINTON,
Chemist, Conn. Agricultural Experiment Station, and Reporter on
Methods of Potash Determination for the Association of Official
Agricultural Chemists for the Years 1896 and 1897 ;
and

H. J. WHEELER,
Chemist, R. I. Agricultural Experiment Station, and Reporter on
Methods of Potash Determination for the Association of Official
Agricultural Chemists for the Years 1894 and 1895.

In view of certain criticisms of the Lindo-Gladding method of potash determination which have appeared of late, notably those by Vogel and Haefcke (*Die Landw. Versuch-Stationen*, 47, 1896, pp. 112—117), we were appointed a committee by the Association of Official Agricultural Chemists of the United States to prepare an article setting forth certain work bearing upon the points involved.

At the regular meeting of the Association in Washington in 1890, Breyer and Schweitzer (U.S. Dept. of Agric., Div. of Chem., *Bull.* 28, 1890, p. 73) called attention to certain comparative determinations made by them, which indicated that the use of sodium chloride was unnecessary. The mean difference in the results with and without sodium chloride amounted to but 0.04 of a per cent, and in but two cases out of fifty was the difference over 0.10 of a per cent. In 1891, Winton (U.S. Dept. of Agric., Div. of Chem., *Bull.* 31, 1891, pp. 148—150) investigated the matter thoroughly, and in tests conducted with commercial potash salts and twenty-four mixed fertilisers showed that the results with and without the addition of sodium chloride were practically identical (compare Fresenius, *Zeit. f. Analyt. Chem.*, xvi., p. 63). The same line of study was further pursued in 1892 by Payne (U.S. Dept. of Agric., Div. of Chem., *Bull.* 35, 1892, pp. 58—61), in co-operation with twenty-two chemists in seventeen different laboratories, working with a variety of substances. As a result of these investigations by the Association, the use of sodium chloride was discontinued (U.S. Dept. of Agriculture, Div. of Chem., *Bull.* 35, 1892, pp. 197—198). This was done, not because inaccurate results were obtained by its use, but in order to abbreviate the method by striking out a useless and cumbersome detail.

Notwithstanding the fact that it had already been abundantly shown that there was practically no difference in the results obtained with and without the use of sodium chloride, Breyer and Schweitzer (*Journ. of Anal. and Appl. Chem.*, vi., No. 8, 1892, p. 477) asserted that its use was a source of error. Their claim that it was unnecessary was merely a reiteration of what had already been shown by the work of the Association.

Aside from the undesirability of employing sodium chloride, the only unfavourable criticism of the Lindo-Gladding method worthy of note which has appeared, is based upon the assumption that a considerable error is introduced through double decomposition resulting from the contact of the ammonium chloride wash solution with potassium platinichloride. Breyer and Schweitzer (*Journ. of Analyt. and Appl. Chem.*, vi., No. 8, 1892, pp. 474—477) and N. Robinson (*Journ. Am. Chem. Soc.*, xvi., 1894, pp. 366, 367) mention errors thus arising, and the two former chemists claim that the amount of the same is considerable. Their conclusions in this respect are based chiefly upon the amount of potassium chloride said to have been recovered from the ammonium chloride wash

TABLE I.—Comparison of Results on Pure Potassium Chloride by the Lindo-Gladding Method and by the Method of Washing with Alcohol only (1894). (0.1 of a grm. of KCl taken in each Determination. Weights in Grms.).

Analyst.	K ₂ PtCl ₆ washed with alcohol only. KCl found.	Results by K ₂ PtCl ₆ washed with alcohol and ammonium chloride solution. (Lindo-Gladding). Results by ammonium chloride more (+) or less (-) than those without.	
		KCl found.	
W. A. Powers, Illinois Station	0.10053 0.10114 0.10084	0.10134 0.10159 0.10147	
Average.. .. .			+0.00063
F. P. Veitch, Maryland College	0.09867	0.09864	-0.00003
J. B. Lindsey, Massachusetts Station ..	0.10112 0.10066 0.10066	0.10082 0.10097 0.10060	
Average.. .. .	0.10082	0.10005 0.10061	-0.00021
W. L. Rossmann, Michigan Station..	0.09957 0.09951 0.09957 0.09933	0.09974 0.09956 0.09971 0.09965	
Average.. .. .	0.09950	0.09967	+0.00017
J. B. Street, New Jersey station . . .	0.09838 0.09868 0.09871	0.09807 0.09838	
Average.. .. .	0.09859	0.09823	-0.00036
B. L. Hartwell, Rhode Island Station ..	0.09960 0.09954	0.09898 0.09913	
Average.. .. .	0.09957	0.09934 0.09915	-0.00042
K. P. McElroy, United States Department of Agriculture ..	0.10000 0.10012 0.10006	0.10015 0.10040 0.10028	+0.00022
R. de Roode, West Virginia Station ..	0.09996	0.09993	-0.00003

solution which had already been employed in washing precipitates of potassium platinichloride. It does not appear, however, that blank determinations were made for the purpose of ascertaining the amount of potash, if any, contained in the ammonium carbonate, ammonium oxalate, and in the 7 grms. of barium hydrate and other reagents employed in the recovery of the potash. In view of the fact that considerable amounts of potash are frequently present in certain so-called chemically pure reagents employed in potash determinations (U.S. Dept. of Agriculture, Division of Chem., *Bull.* 43, 1894, pp. 21, 22; also *Bull.* 47, 1895, pp. 16, 17; and *Bull.* 49, 1896, pp. 30—32), it is obvious that a large part of the potash said by Breyer and Schweitzer to have been recovered from the wash solution may have been derived from their reagents. It is obvious, therefore, that their results, instead of serving as a proper basis for the criticism of a method, are themselves open to serious criticism.

The ammonium chloride wash solution experimented with by Breyer and Schweitzer was the same as that employed by the Association, the preparation of which is described in a succeeding footnote. That a great amount of double decomposition may result with this solution, under exaggerated conditions, has been shown by the work of the Association (U.S. Dept. of Agric., Div. of Chemistry, *Bull.* 43, 1894, pp. 23, 24; and *Bull.* 47, 1895, p. 22) during 1894 and 1895. This fact alone furnishes

TABLE II.—Comparison of Results on Pure Potassium Chloride by the Lindo-Gladding Method and by the Method of Washing with Alcohol only (1895). Weights in Grms.

Analyst.	K ₂ PtCl ₆ washed with alcohol only.			K ₂ PtCl ₆ washed with alcohol and ammonium chloride solution (Lindo-Gladding).		
	Taken. KCl.	Found. KCl.	Error. KCl.	Taken. KCl.	Found. KCl.	Error. KCl.
W. A. Powers, Illinois Station	{			0'3976	0'3954	-0'0022
				0'3872	0'3871	-0'0001
B. L. Hartwell, Rhode Island Station*. .. .	{	0'4776	+0'0004	0'4534	0'4532	-0'0002
		0'4458	+0'0005	0'4799	0'4797	-0'0002
		0'4665	+0'0003			
		0'5437	+0'0005			
G. Wm. Gray, West Virginia Station .. .	{	0'5114	-0'0004	0'5028	0'5002	-0'0026
				0'5080	0'5049	-0'0031
F. P. Veitch, Maryland College .. .	{	0'5004	-0'0002	0'5043	0'5058	+0'0015
		0'5666	+0'0019	0'5149	0'5163	+0'0014
		0'5005	+0'0022			
W. H. Allen, North Carolina Station.. .	{			0'2987	0'2989	+0'0002
				0'2987	0'2988	+0'0001
				0'2987	0'2985	-0'0002
J. B. Lindsey, Massachusetts Station.. .	{	0'4978	+0'0009	0'4978	0'4985	+0'0007
		0'4978	+0'0008	0'4978	0'4979	+0'0001
W. G. Brown, United States Department of Agriculture	{	0'6479	+0'0027	0'5550	0'5559	+0'0009
		0'5731	+0'0018	0'5557	0'5560	+0'0003
		0'4796	+0'0002			
J. P. Street, New Jersey Station.. .	{	0'4978	+0'0003	0'4978	0'4996	+0'0018
		0'4978	-0'0001	0'4978	0'4993	+0'0015
				0'4978	0'4985	+0'0007
E. P. Stone, New Hampshire Station.. .	{	0'5022	+0'0021	0'5022	0'5043	+0'0021
		0'5883	+0'0019	0'5883	0'5902	+0'0019
A. L. Winton, Connecticut Station.. .	{	0'4500	+0'0008	0'5011	0'5010	-0'0001
		0'5004	+0'0009			
		0'5331	+0'0007			
		0'5310	+0'0003			

* These results have been corrected for an error in the original table.

no suitable basis for the condemnation of the method, provided the amount of error arising in the ordinary course of analysis is practically insignificant.

During the years 1894 and 1895 (U.S. Dept of Agric., Div. of Chem., *Bull.* 43, pp. 17-25; *Bull.* 47, pp. 14-23), one of us (H. J. W.) conducted comparative determinations to learn whether washing with ammonium chloride solution, according to the Lindo-Gladding method, introduced an appreciable error. The analytical work was carried out with the co-operation of the analysts of the Association of Official Agricultural Chemists of the United States, nine taking part during the first year and ten during the second year, no two of them working in the same laboratory or with the same reagents.

In 1894, portions of 4 grms. each of pure potassium chloride were made up to 1000 c.c. with water, and 25 c.c. of this solution, representing 0.1 of a gm. of the salt, were taken for each determination. As the salt contained a small amount of water, which in the portions sent to the different analysts varied from 0.06 to 0.16 per cent, the results have in each case been calculated to correspond with 0.1 of a gm. of the dry material (see Table I.).

The determinations carried out in 1895 were made on solutions of 50 c.c. each, containing from 0.3 to 1.0 gm. of pure potassium chloride, thus exposing to the action of the ammonium chloride solution three to ten times as much potassium platinichloride as in the preceding year.

The salt used was from a new lot containing uniformly 0.45 per cent of moisture; the weights of KCl taken, as given in Table II., represent, however, the dry salt.

In the determinations of both years, each solution of the pure salt was evaporated to a paste with an excess of platinum solution. The residue, after cooling, was stirred for twenty minutes with 30 c.c. of 80 per cent alcohol (sp. gr. 0.8645) and the liquid decanted on a weighed Gooch crucible. The washing was further continued by decantation, using 10 c.c. portions of alcohol and stirring from two to three minutes after each addition.

In some cases the salt, without further washing, was dried at 100° C. and weighed, in others it was first washed with six portions of ammonium chloride solution* of 10 c.c. each, and finally with 80 per cent alcohol, according to the Lindo-Gladding method.

Blank determinations were made by both methods on the reagents.

From the analytical data secured in these determinations, which were given in full in *loc. cit.*, Tables I. and II. have been prepared.

In calculating the results, the total weight of potassium platinichloride obtained in each determination was first corrected by deducting the average weight obtained by the same analyst from the reagents.

* A solution of 100 grms. of ammonium chloride in 50 c.c. of water, which had been shaken for from six to eight hours with 5 to 10 grms. of pulverised potassium platinichloride, allowed to settle over night, and filtered.

For the conversion of K_2PtCl_6 to KCl the factor 0.3069 (Pt 195.0, K 39.11, Cl 35.45) was employed, for reasons stated later.

A careful study of the results given in the tables will make it evident that the potassium platnichloride was very slightly, if at all, acted upon by the ammonium chloride solution. Finkener (*Pogg. Ann.*, cxxix., p. 697) found that a saturated solution of ammonium chloride at 22° C., when in contact with finely-divided potassium platnichloride for one hour, decomposed but 0.27 per cent of the latter. Since the Lindo-Gladding method, at least as applied to the potash salts, secures the double salt in a granular condition, which, according to Finkener, is not so rapidly acted upon, and since, operating with a Gooch crucible, the ammonium chloride solution is not in contact with the double salt longer than two or three minutes, errors from this source would not be expected to be, and, as numerous results show, are not appreciable. There are few, if any, analytical methods where the precipitate is absolutely insoluble in the wash liquid, and it is absurd to discriminate against the Lindo-Gladding method for theoretical reasons which have no practical significance.

(To be continued).

ON THE SEPARATION OF ALUMINIUM.

By RUDOLF L. LEFFLER, F.C.S.

The "Neutralisation" of Aluminium Chloride.

"NEUTRALISATION" is placed in commas because it must not be taken to mean only the converting of free acid into salts. Although in certain connections a special meaning is well enough understood, one cannot help feeling that the word is not a happy one. It must here be taken to mean not only the "neutralisation" of the acid, but also the continued precipitation of aluminium or ferric hydrate until its undecomposed chloride can no longer dissolve it. In reference to ferric solutions, to "neutralise" frequently carries this special meaning, because it is the state into which such solutions are converted prior to the familiar separations with alkaline salts. The degree to which ferric hydrate may be precipitated and re-dissolved is influenced by the amount of alkaline salts in solution. They tend to hasten the point at which a permanent precipitate forms; but with moderate amounts—and by this I mean such as may be due to the usual amounts of free acid—the proportion between the ferric chloride and the ferric hydrate it holds in solution is generally agreed to be about $Fe_2Cl_6 : 7Fe_2(HO)_6$. With solutions of ferric nitrate the proportion may go up to $Fe_2(NO_3)_6 : 8Fe_2(HO)_6$. It appears also to be indifferent to a solution of ferric chloride, whether it be neutralised in the cold or near boiling-point; the successive precipitates dissolve more rapidly in the latter case, but the proportion $xFe_2Cl_6 : yFe_2(HO)_6$ is the same either way.

The foregoing facts are already well known and utilised daily, but the analogous behaviour of solutions of aluminium does not seem to be as well known.

I find, on turning to the Dictionaries of Watts and Comey, that the above-mentioned behaviour of ferric hydroxide is prominently announced, and respecting the hydroxide of aluminium I find it stated to be insoluble in $FeCl_3$, soluble in $Fe(NO_3)_3$, $Cr(NO_3)_3$, $Bi(NO_3)_3$, $NaC_2H_3O_2$, &c.; but nowhere do I find it suggested even that it is soluble in its own chloride or other salts. All this notwithstanding it is common to find an instruction to the effect that solutions of aluminium should be neutralised; but in view of the ambiguous meaning of the word it is not easy to say exactly what is meant. Allotting to it, however, the more special meaning we associate with ferric solutions, it remains to be pointed out that the

operation is usually performed in the cold, either expressly so (Blair, "Chem. Anal. Iron," p. 232) or in anticipation of the next step, viz., the addition of acetate, which according to a mistaken notion "should be added to the metallic solution when cold, and the whole then heated together" (Crookes, "Select Methods," p. 227). Such conditions make the complete "neutralisation" of aluminium solutions impossible.

With ferric chloride the colour-change tells when the free acid is neutralised, and the deepening colour indicates the progress of the "neutralisation"; but with aluminium chloride such observations cannot be made; one has to depend entirely on the appearance of a permanent precipitate. Quarter of a gram of aluminium in solution as Al_2Cl_6 would require 27.4 c.c. normal alkali to precipitate it. If we take such a solution (0.25 grm. Al) in the cold, and add, say, soda carbonate from a burette, we shall find the precipitate dissolve readily enough at first, but, after the addition of a few c.c., precipitates are formed which according to one's knowledge of ferric solutions might be called permanent. To quote the regular formulæ, "they do not disappear on vigorous stirring." When about 21.9 c.c. has been added ($= Al_2Cl_6 : 4Al_2(HO)_6$), they go neither with vigorous stirring nor prolonged standing. In fact, they may stand undissolved for a day, but on warming this long-stood turbid solution it dissolves up crystal clear. If we repeat the whole operation with a warm or hot solution of Al_2Cl_6 , no permanent turbidity forms until 25.2 c.c. has been added. This proportion is approximately $Al_2Cl_6, 11.5Al_2(HO)_6$. On diluting and boiling the aluminium is not precipitated, so that the Herschel or Schwarzenberg separation would appear to be inapplicable to aluminium. Instead of deepening, a decided turbidity would seem to clear somewhat after this operation. If more carbonate be added, the turbidity—or perhaps better the opalescence—deepens, but no precipitate can be detected; in this way the proportion may reach $Al_2Cl_6, 23Al_2(HO)_6$, wanting only an additional 1.20 c.c. to bring down the whole 0.25 grm. metal. Nitrates have less influence in lowering the "neutralisation" point than chlorides. This difference is less marked with ammonia salts than with soda salts.

The "neutralisation" of metallic solutions is a valuable device in effecting the separation of closely-allied elements, because it enables one to use very small amounts of a precipitant which used in excess tends to precipitate those metals one wishes to leave in solution. To explain imperfect acetate separations by saying that the iron, for example, carries down portions of the other metals is wrong, because it associates the error with the precipitation and makes it seem a necessary accompaniment, whereas the iron *must be* precipitated, but no portion of the manganese, say, needs be. The degree to which a solution has been neutralised, and the excess of acetate used, has been shown in a previous communication from this laboratory to be responsible for the better or worse separation of iron from metals of its own group and from copper. There are two current ideas respecting acetate separation of aluminium, founded no doubt on experience. These are:—

- I. That aluminium has more tendency than iron to carry down associated metals.
- II. That the aluminium itself is not completely precipitated, and therefore it is necessary to examine filtrates, &c.*

In the following section we seek to explain these difficulties and to minimise them:—

The Precipitation of Al_2Cl_6 .

Iron may be precipitated in fair accordance with the equation—



* See Fresenius, "Quantitative Analysis," pp. 439–450; "Select Methods," p. 199; Arnold's "Steel-works Anal.," p. 245; Dittmar, "Quant. Anal.," p. 74.

* Doubtful.

Does an analogous equation represent the precipitation of Al_2Cl_6 ? Experiment shows that it does not, for if 0.25 grm. of aluminium as chloride be taken instead of precipitating with approximately 4 grms. crystallised soda acetate it will behave in hot solutions as follows:—Add 1 grm. acetate at a time. The second grm. makes the solution opalescent, and this opalescence increases until 6 grms. have been added. Seen casually the solution appears to hold a precipitate, but examined closely no form or body of a precipitate can be seen. The next grm. precipitates the aluminium, and enables us to obtain a clear sparkling filtrate, but on adding ammonia it is seen that *all* the aluminium was not precipitated.

Supposing only 4 grms. of the acetate to have really combined with the aluminium, the presence of alumina in the filtrate may be due to the decomposition of the $Al_2(C_2H_3O_2)_6$ with formation of acetic acid capable of dissolving $Al_2(OH)_6$, and to the excess of soda acetate in which $Al_2(OH)_6$ is said to be soluble. Further, according to the explanation of Brearley (CHEM. NEWS, lxxvi., 223), the necessary excess of acetate would also account for the more incomplete separation of associated metals expressed "by the greater tendency of aluminium to carry down other metals."

Precipitation from Total Hydrates.

"Neutralised" to faint opalescence in *hot* solutions (Na_2CO_3 or NH_4HO), being careful to add at a time only so much as readily dissolves, or, in keeping with familiar experience, the digested $Al_2(OH)_6$ does not dissolve easily, and decomposed the remaining Al_2Cl_6 with the smallest practicable quantity of acetate. It might be anticipated that, having now to decompose only about one-tenth of the former Al_2Cl_6 , it could be done with one-tenth of the former acetate, but a considerable excess is needed here also—hence our inability to completely eliminate the errors in every case. It was found that 0.25 and 0.5 grm. Al, completely "neutralised," required 2.25 and 3.75 grms. respectively of soda acetate, or its equivalent of ammonium acetate, to completely precipitate the aluminium; the filtrates were free from alumina when hot, but if allowed to cool during filtering then alumina was always present.

The necessity for the use of these unexpected large amounts of acetate in the precipitation of the "neutralised" solution tells very much against this means of separating aluminium from other elements. Separations from nickel were tried, and minimum amounts of acetate used. It is necessary to keep solutions very hot during filtration, so as to avoid the re-solution of the precipitated $Al_2(OH)_6$. The results were always 1 or 2 per cent too low; moreover, the small amounts of alumina which, with all care, sometimes pass into the filtrate, tend to give low results when the nickel is estimated cyanometrically—a difficulty which at present I have not learned how to completely avoid. From copper the separations are even worse.

The only separation worthy to be called quantitative, and deserving to be noticed, is that from manganese, which it may be recorded is similarly indifferent to moderate excesses of acetate in its separation from iron.

Separations with Sodium Phosphate.

Ni from Al.—Twenty c.c. of a saturated solution of this reagent is required to completely separate half a grm. of aluminium from its "neutralised" chloride. Varying quantities of precipitant, with and without acetic acid, were tried. In each case 0.5 grm. Al and 0.10 grm. Ni was taken; volume of solution, 1 litre; fractional filtration; and the Ni estimated by titrating with KCN (CHEM. NEWS, lxxvi., 49). The precipitates settled quickly, more especially with the larger quantities of acetic acid, and with a little care the filtrate came through the paper (dry ribbed) as quick as one could pour on; they were free from alumina. The figures given are percentage recovery.

Satd. sol. sodium phosphate. C.c.	Acetic acid. C.c.			
	0.	10.	20.	30.
20	89.40	—	—	—
30	45.36	100.0	—	—
50	—	81.0	97.05	99.72
70	—	62.36	91.94	96.41

It appears from these results that the separation can be made only within narrow limits, and that these depend solely upon the proportion between the phosphate and the acetic acid. The separation of copper and zinc have been experimented with on these lines, but the results were not good.

It must be owned that the behaviour of these completely "neutralised" solutions have not come up to our expectations of them; still we hope to have shown on what grounds "the greater tendency of aluminium than iron to carry down associated metals" rests, and how the most satisfactory acetate separations may be made.

The Laboratory,
Messrs. T. Firth and Son, Lim., Sheffield.

ON IODIDE OF GLUCINUM.

By P. LEBEAU.

THE formation of iodide of glucinum by the action of iodine on the metal was first pointed out by Wöhler (Pogg. Ann., vol. xiii., 1828), and later by Debray (Ann. de Chim. et de Phys., Series 3, vol. xlv., p. 24, 1855). They describe this compound as a white sublimate, very volatile according to one of them, but less volatile than the chloride according to the other. No work respecting the study of this body has been published since. Further, the preparation of the metal seems to have been sufficient obstacle to prevent further research.

We have been able during our research on the properties of carbide of glucinum, to find a method enabling us to obtain with the greatest ease a considerable quantity of definite crystallised iodide of glucinum. In fact, the vapour of iodine carried by a current of hydrogen, or, better still, dry hydriodic acid, attacks the carbide of glucinum at about 700°, producing a very pure iodide of glucinum. To prepare this iodide with the least trouble, we arrange a tube of Bohemian glass, of 20 or 25 m.m. diameter, on a small gas furnace. The tube should be at least twice as long as the furnace. Five or six grms. of powdered carbide of glucinum are placed in a porcelain boat, or even in the tube itself. A current of hydriodic gas is passed, and the tube then heated to a cherry-red heat. The attack of the carbide goes on in a regular manner, the iodide distils off and condenses in the cold part of the tube in a felty mass of crystals.

When the carbide is very pure, we obtain a perfectly white iodide, which must be collected in a vessel filled with dry carbonic acid gas. If it is of a yellow colour, which is caused by a small quantity of iodide of iron, it suffices to sublime it in a current of dry carbonic acid; the iodide of iron is carried off from the commencement, after which the iodide of glucinum is sublimed in very beautiful crystals.

Analysis has shown us that this body answers to the formula GI_2 or $GI_{1.6}$, according to whether we take the atomic weight as 9.08 or 13.8.

				Calculated for $GI_{1.6}$
Iodine, per cent	..	96.46	96.37	96.49
Glucinum	..	3.49	3.52	3.47
		99.95	99.89	99.96
				99.99

Iodide of glucinum exists in the form of colourless crystals, very easily altered in moist air. Its density at 15° is very close to 4.20. It melts at about 510°, but sublimes

considerably before melting. Its boiling point is somewhere between 585° and 595° ; it should apparently therefore be easy to determine its vapour density. We shall return to this subject later on. Iodide of glucinum is insoluble in benzine, toluene, and essence of turpentine, and slightly soluble in sulphide of carbon.

Water reacts violently on iodide of glucinum, giving a soluble hydrated iodide. This property renders the handling of this body a matter of some delicacy, as the least trace of water partially decomposes it. When melted it is much less easily altered, and it is in this form that we have studied its principal properties.

It can be distilled in a current of dry hydrogen without any alteration. Chlorine and bromine decompose it, forming the corresponding compounds, with the liberation of iodine. Fluorine attacks it, forming fluorides of iodine and glucinum.

Fluorine and chlorine act in the cold with incandescence.

Cyanogen reacts on iodide of glucinum below a red heat, and forms a less volatile white substance, producing with water a clear solution having the characteristics of a cyanide. Until now it had not been possible to obtain any cyanic combination of glucinum.

Heated in oxygen, iodide of glucinum takes fire below a red heat. Its vapour burns on contact with air. Sulphur transforms it into a fixed sulphide at a bright red heat; it is decomposable by water.

This experiment clearly shows that there is a sulphurised compound of glucinum. It may be remembered that Debray and Frémy stated that they had not been able to prepare sulphide of glucinum by the ordinary methods.

In a current of nitrogen the iodide distils without changing its composition or appearance. The vapour of phosphorus, on the contrary, destroys it, and forms a combination of phosphorus and glucinum.

Sodium reduces the iodide of glucinum at about 350° ; potassium and lithium also react with incandescence at about the same temperature. Alkaline iodides of glucinum are formed. At about 450° magnesium gives an iodide of magnesium and glucinum; this reaction is an important one. Aluminium, silver, copper, and mercury are all without action below a temperature which softens the glass.

Sulphuretted hydrogen does not react at the ordinary temperature, but if it is gently heated hydriodic acid is formed, and a fixed white substance remains, analogous to the sulphide obtained by the action of sulphur. Ammonia gas is absorbed very rapidly in the cold, forming a white powder, much less alterable than the iodide, and giving by analysis the formula $2\text{GII}_2, 3\text{NH}_3$. On gently heating, a further absorption of ammonia takes place, and the substance melts into a colourless liquid, crystallising on cooling. A prolonged contact gives further rise to another body even richer in ammonia.

Iodide of glucinum reacts on a large number of organic compounds.

It dissolves in the alcohols perfectly free from water, without appreciable heating. With absolute alcohol a crystalline compound is obtained. With ether it also forms a compound, slightly soluble in an excess of ether.

Like the iodide of aluminium, it has no action in the cold on tetrachloride of carbon. It is equally without action on the chloride C_2Cl_4 . Acetic anhydride, chloral anhydride, and a very large number of oxygenated organic compounds, give energetic reactions.

Finally, the ammoniacal compounds and the organic bases, notably aniline and pyridine, produce crystallised compounds with iodide of glucinum.

Conclusions.—To sum up; by the action of hydriodic acid on carbide of glucinum we have obtained an iodide of the formula GII_2 , in beautiful transparent crystals. This new iodide is very active, it reacts on a large number of bodies, and serves for the easy preparation of new compounds of glucinum, such as the phosphide, the

sulphide, and the cyanide. It also unites with facility with organic compounds.—*Comptes Rendus*, vol. cxxvi., No. 18.

NEW METHODS OF DETECTING AMMONIA IN A GASEOUS ATMOSPHERE.

By G. DENIGÈS.

THE detection of ammonia gas in a gaseous atmosphere has been made extremely easy by the use of Nessler's solution, introduced into this atmosphere on a glass rod.

However, the simultaneous presence of amines of the fatty series deprives the mercurio-potassic iodide, in alkaline solution, of its special power; monomethylamine and monethylamine give, in fact, coloured precipitates with this reagent, and the secondary methylic and ethylic amines—to mention only those best known—also form insoluble mercuric compounds.

For these reasons it has appeared to us useful to make known a more specific reaction for ammonia, of which we have made use for some time, as well as reactions which are less characteristic, though interesting, on account of their great sensitiveness. The principle of neither of them is new, but the method of application allows us to qualify these two processes for the detection of ammonia gas, as new.

The first of these consists in plunging the extremity of a glass stirring rod, wetted with hypobromite of soda, into the gas to be tested; on contact with ammonia gas, the wet part of the glass rod gives off a large number of small bubbles of nitrogen gas, so small that they appear as a white sheath round the extremity of the stirrer. At the same time the hypobromite loses its colour.

The property of behaving in this manner to hypobromite of soda belongs *only* to ammonia; in fact, with this reagent the primary amines give a yellowish precipitate, while the other fatty amines cause no notable phenomenon at all.

The two other reactions which we wish to describe belong, like that of mercurous nitrate, as much to the fatty amines as to ammonia gas.

The one is the invert reaction of formol: it is obtained by submitting a drop of commercial formol to the contact of ammoniacal vapours, and then plunging it into 1 c.c. of bromine water acidulated with a drop of acetic acid. In this manner we immediately obtain a cloudiness or a yellow precipitate, produced by a bromine derivative of hexamethylene tetramine.

The other is based on the intense carmine colouration which one drop of an aqueous solution of hæmatoxyline, or of extract of logwood, takes in contact with even very small quantities of ammonia gas.

From the point of view of their degree of specific action we may class the principal reagents of ammonia and the fatty amines in the following manner:—

1. Hypobromite of Sodium.

This body gives off bubbles of nitrogen gas under the influence of ammonia. It gives insoluble yellow compounds with the primary fatty amines. It does not react sensibly on the secondary and tertiary amines.

2. Nessler's Solution.

Gives a Kermes-red precipitate with ammonia, a yellow precipitate with monomethylamine, and a white precipitate with monethylamine. This latter precipitate rapidly becomes yellow if the reagent is in excess, and regains its white colour with an excess of amine. With dimethylamine and diethylamine it gives a white precipitate; with the latter body the precipitate is soluble in an excess of amine.

Finally, with trimethylamine, and above all in an excess of amine, it gives a white precipitate, rapidly becoming brown.

3. *Mercurous Nitrate, Hæmatoxyline, Formol.*

The first of these bodies in solution becomes black on contact with ammoniacal vapours or fatty amines.

The second takes a carmine colour, and the third gives hexamethylene tetramine, which forms a yellow precipitate with bromine water acidulated with acetic acid.—*Bull. de la Soc. de Pharmacie*, March, 1898.

ON THE ABSORPTION OF NITRIC OXIDE
BY FERROUS SALTS.

By M. THOMAS.

It has long been known that all ferrous salts in solution absorb dioxide of nitrogen. The classic work of Péligré (*Ann. Chim. Phys.*, Series 2, xiv., 17, 1803), and the more recent research of M. Gay ("Thèse de Doctorat," 1885), go to prove that we obtain quite definite compounds in the form of solution, with a dissociation-tension at the ordinary temperature. By the evaporation of nitrous solutions, even in an atmosphere of binoxide of nitrogen, we certainly obtain black crystals, but these crystals only contain traces of nitric oxide.

As for the quantity of gas absorbed, the researches of M. Gay led him to the conclusion that the quantity of binoxide of nitrogen absorbed by the salts of peroxide of iron is:—

1. Independent of the nature of the salt.
2. Independent of the degree of dilution of the solution.
3. Proportional to the minimum weight of iron that it contains.
4. A function of the temperature.

This absorption is, 1 molecule of nitric oxide for 2 atoms of iron, at a temperature above 12.5° , and 2 molecules of the gas for 3 atoms of iron below that temperature.

I shall show successively that—

1. All ferrous salts, without exception, possess the same property of absorbing nitric oxide in aqueous solution. (Verification of M. Gay's law).

M. Gay studied the absorption by the chloride, the sulphate, and the ammoniacal sulphate. The absorption takes place in an analogous manner with the most varied salts, such as the chlorate, perchlorate, bromide, bromate, iodide, sulphite, hyposulphite, hyposulphate, &c.

2. All ferrous salts retain this property when another solvent is substituted for water.

The absorption of binoxide of nitrogen by ferrous salts dissolved in different solvents is an important point to know, if we wish to form any hypothesis on the different behaviour of solid or dissolved ferrous salts with nitric oxide. In fact, if we take the aqueous solution of a salt and evaporate it, either *in vacuo* or by any other means, the ferrous salt is left, generally well crystallised, in the form of a hydrate. We are thus brought to the opinion that, in aqueous solutions, the ferrous salt must be present in the anhydrous state, but in combination with water. From this it follows that the absorption of nitric oxide might be caused by the existence of this hydrate in the dissolved state.

I was therefore persuaded to try whether the absorption would take place by substituting other solvents for water.

Solvent used: Alcohol.—Graham (*Phil. Mag.*, iv., 231 and 266) used an alcoholic solution, and showed that nitric oxide was still absorbed by ferrous chloride. He even showed that the absorption was greater than in an aqueous solution, and that 1 atom of iron was capable of absorbing 1 molecule of binoxide of nitrogen. Further, all the gas was given off, either by evaporation *in vacuo* or by spontaneous concentration by leaving the solution at the ordinary temperature. It follows, therefore, that

the alcoholic nitrous solution has properties entirely comparable with those of the aqueous solution.

Not only the chloride, but all the salts tried by me behaved in the same manner; for instance, the bromide and the iodide. However, as Graham has shown that the ferrous chloride was able to absorb vapour of alcohol as well as aqueous vapour, and as I, on the other hand, have shown that the chloride and bromide react chemically on alcohol, giving, without doubt, some double compounds analogous to the hydrates, it might here again be objected that the absorption might be caused by these compounds in solution, and not by the ferrous salt itself.

Solvent used: Ether.—If we reduce a solution of ferric chloride or bromide by binoxide of nitrogen, the ferrous salt formed remains in solution, and the solution is strongly blackened by the quantity of nitric oxide it retains. By evaporation the solution becomes syrupy, and voluminous crystals are seen, which the microscope shows to be in the form of prisms of a bright red colour forming macles; this represents the product of the reaction of the ferrous chloride on the ether. By using acetic acid and bromide of ethylene, which leaves by evaporation the anhydrous ferrous salt, we can, on the contrary, show that the absorption of gas is really due to the ferrous salt itself.

Solvents used: Acetic Acid, Bromide of Ethylene.—The acetic acid used was crystallised at 16.25° , and was then sufficiently pure for the experiments.

The absorption of nitric oxide takes place in acetic solution, the same as in aqueous, alcoholic, or ethereal solution.

Bromide of ethylene behaves in every respect just like acetic acid.

3. The absorption of nitric oxide is variable according to the solvent used.

This result is found from the figures given for the absorption by ferrous chloride in aqueous solution by M. Gay, and in alcoholic solution by Graham.

Absorption in Aqueous Solution (Gay).

	For 56 of Iron.	Per cent.
From 4° to 12° ..	19.48	34.78
" 16° to 22° ..	15.00	26.78
To 25°	10.80	18.92

Absorption in Alcoholic Solution (Graham).

The information given in Graham's papers shows the proportion of binoxide of nitrogen to be greater than 50 per cent.

Furthermore, I have repeated these experiments, and determined the quantity of gas absorbed exactly. The absorptions were determined volumetrically; the following was the method employed:—Into a graduated tube resting in the mercury trough, a certain volume of ferrous chloride of known strength was introduced. By the aid of a second graduated tube a known volume of binoxide of nitrogen was added. The gas was prepared by the action of nitric acid on mercury; the use of copper and nitric acid gives not only protoxide of nitrogen as an impurity, but also nitrogen which cannot be got rid of. I will now give a few figures:—

- I. 12.2 c.c. of a solution containing 4.6 grms. of iron per 1000 c.c. absorbed 21.5 c.c. of nitric oxide at a temperature of 17° .
- II. 12.5 c.c. of the same solution absorbed 23.4 c.c. at a temperature of 15° .

This gives for NO in percentage of iron:—

- I. At 17° 51.37 per cent.
- II. At 15° 54.57 "

These figures, it will be seen, are quite comparable with those obtained by Graham.

Remarks.—The gaseous volumes measured were brought to 0° . To determine the exact volumes not absorbed the readings were made at a pressure of 760 m.m., augmented by the pressure of the maximum tension of the solvent.

This tension, negligible in the case of water, is not so in the case of alcohol.

In a succeeding paper I shall discuss the absorption of nitric oxide by ferrous bromide and ferrous iodide.—*Bull. Soc. Chim.*, Series 3, vol. xix.-xx., No. 10.

ON THE CONDITION OF OXIDATION OF MANGANESE. PRECIPITATED BY THE CHLORATE PROCESS.*

By F. A. GOOCH and MARTHA AUSTIN.

HANNAY (*Journ. Chem. Soc.*, xxxiii., 269), who was the first to propose the precipitation of manganese from its solution in nitric acid by the use of potassium chlorate, states that precipitation is complete, but that the oxide produced is not of constant composition. While, therefore, precipitation by this method serves an excellent purpose in separating manganese from other substances, it was Hannay's opinion that reliance cannot be placed upon the determination of the oxygen value of the oxide to estimate the manganese. Beilstein and Jawein (*Ber. d. Chem. Gesell.*, xii., 1528), who proposed the same method, subsequently, regarded the precipitate as the oxide MnO_2 . Hannay's reaction was developed, independently, by Hamps (*Chem. Centralblatt.*, 1885, 714) and Ford (*Trans. Inst. Am. Min. Engineers*, ix., 347) into the method which is now known as the "chlorate process" for the estimation of manganese. The discussion of the exact condition of the precipitated oxide was very active ten years ago, and occasional echoes of it are heard at the present day; and yet, in all this discussion we find no account of an adequate test of the process upon an exact amount of a salt of manganese known to be pure. The discussion for the most part has centred about the degree of oxidation of the precipitate, but there is obviously another condition to be taken into account, viz., the possibility of the mechanical inclusion of the comparatively insoluble chlorate in the precipitated oxide. As to the existence of the latter source of error we have had in the course of our work very strong affirmative evidence, the apparent condition of oxidation of the precipitate being sometimes so high as to be otherwise inexplicable. This difficulty does not occur, however, when a more soluble chlorate is chosen to do the work of oxidation, and we have found quite as convenient and much safer the substitution of sodium chlorate for the comparatively insoluble potassium chlorate. Besides, the rapidity with which the sodium chlorate is decomposed makes its use an advantage.

With regard to the completeness of the precipitation, our experience has shown that with due precaution the method is practically perfect. Thus, after boiling manganous nitrate (free from chlorides and sulphates) with strong nitric acid (85 c.m.³) and sodium chlorate (5 grms.) for five minutes, adding subsequently 15 c.m.³ of the nitric acid and a few crystals more of the sodium chlorate, and discontinuing the heating as soon as the liquid again boils, the insolubility of the manganese is so great that no more than insignificant traces may be recovered from the filtrate after cooling, filtering upon asbestos, and washing with water. The test for manganese in the filtrate and washings was made after evaporation and solution of the residue in distilled water by treating the hot solution with bromine and ammonia. In the first division of the table below are results obtained by treating the manganese precipitated from the filtrate with potassium iodide and sulphuric acid, the iodine set free being determined by sodium thiosulphate. In the second series, the precipitated manganese dioxide was reduced by a known amount of decinormal arsenious acid and the

amount remaining unoxidised was estimated by titration with iodine in the presence of acid potassium carbonate.

TABLE I.

MnSO ₄ taken. Grm.	Mn found by KI treatment in filtrate. Grm.	Mn found by As ₂ O ₃ treatment in filtrate. Grm.
0.3361	none	—
0.3361	none	—
0.3361	0.00006	—
0.3361	0.00005	—
0.3361	0.00002	—
0.3361	0.00008	—
0.3361	none	—
0.4128	—	0.00003
0.4128	—	0.00003
0.4128	—	trace
0.4128	—	trace

It will be seen that in no case did the manganese which escaped precipitation—that which corresponded to the iodine freed or the arsenious acid oxidised—exceed 0.0001 grm. Plainly, this modified method of handling the chlorate process may be trusted to precipitate the manganese with gratifying rapidity and approximation to completeness. Our experience has shown plainly that prolonged boiling results in a considerable loss of manganese (from 0.0010—0.0030 grm.). This, we think, is due to the solvent effect of the lower oxides of nitrogen naturally produced (as is always the case in boiling nitric acid) after the chlorine dioxide has been thoroughly expelled. An excess of the chlorate at the end of the boiling seems to be essential and a slight yellow colour in the solution, due to chlorine dioxide, is a favourable indication rather than the reverse. We find it best to filter the undiluted nitric acid, under pressure, upon asbestos on a perforated cone with a filtering surface of about 40 sq. cm. The dilution of the nitric acid before filtration tends to produce some solubility of the manganese, and the loss then introduced, though trifling if the filtration is rapid, may be considerable if the process of filtration is prolonged, as is the case in the method approved by the Verein der Deutschen Eisenhütteleute (Von Reis, *Zeit. Angewandte Chem.*, 1891, 376).

Our experiments upon the chlorate process have been made with manganous chloride prepared as detailed in a former paper, viz., by boiling manganous chloride with manganous carbonate, precipitating the filtered solution with ammonium sulphide, dissolving the washed manganous sulphide in dilute hydrochloric acid, precipitating the solution thus obtained with sodium carbonate (after boiling out hydrogen sulphide), dissolving the greater part of the manganous carbonate (thoroughly washed by repeatedly boiling it in successive portions of water) in the least amount of hydrochloric acid, and boiling the solution thus obtained with the remainder of the pure carbonate and filtering. The standard of the solution thus prepared, neutral and probably very pure, was fixed by evaporating definite portions with sulphuric acid and weighing the residue as the normal sulphate, in accordance with the procedure outlined in a former paper (*Am. Journ. Science*, IV., v., 209; *CHEM. NEWS*, lxxvii., 255).

Any method by means of which the oxidising power of the higher oxygen compounds of manganese is discoverable may, obviously, be employed to determine the condition of the manganese precipitated in this chlorate process. Convenient processes for the determination of the available oxygen in the higher oxides of manganese are the iodometric methods of Bunsen and Pickering. Bunsen's method is applicable to any of the higher oxygen compounds of manganese—though somewhat inconvenient, because it involves the distillation of the chlorine liberated by the action of strong hydrochloric acid upon the substance and its collection in potassium iodide, the iodine thus set free being estimated by standard thiosul-

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science* vol. v., p. 260.

phate. According to Pickering's method (*Four. Chem. Soc.*, xxxvii., 128), the higher oxide is treated immediately with potassium iodide and hydrochloric acid, and the iodine liberated is estimated by sodium thiosulphate. Plainly, the latter method is limited to the treatment of the less refractory or more finely comminuted oxides, and it fails in the presence of ferric salts and all other substances capable of liberating iodine from the acidified iodide.

Still another general iodometric method for determining the oxygen value of the higher oxides of manganese is suggested by Deshayes's titration of permanganic acid in nitric acid by means of standard arsenious acid (*Bull. Soc. Chim.*, xxix., 541). Our experience in following out this idea shows that the precipitated oxides of manganese, as well as the soluble permanganate, may be easily reduced with the aid of gentle heating by arsenious acid in the presence of sulphuric acid, and that the determination of the excess of arsenious acid by titration with iodine after neutralisation of the free sulphuric acid by an alkaline carbonate gives exact data for estimating the oxidising power of the manganese compound. We found, however, that if the iodine is allowed to come into contact with the manganous carbonate thrown down by the alkaline carbonate, as is inevitable for at least short intervals during the titration of the arsenious acid in presence of the floating carbonate, the danger arises of more or less re-oxidation of the manganous carbonate by the iodine and the consequent introduction of error. Fortunately the difficulty may be obviated by adding to the solution, while still acid, enough tartaric acid or alkaline tartrate, to prevent the precipitation of the manganese in the subsequent neutralisation by the bicarbonate.

(To be continued).

THE SEPTIC TREATMENT OF SEWAGE.

THE Local Government Board have decided to give their approval to the scheme submitted to the Exeter City Council by Mr. Cameron, the City Surveyor, for disposing of the sewage of Exeter by the Septic system at an estimated expenditure of £40,000.

In their letter to the Exeter City Council, the Local Government Board state:—"The Board have directed their inquiries, with the view of determining two questions. In the first place, they desired to ascertain whether the final effluent obtained by the Septic process was uniformly of such a character as to permit of its discharge into the river without further treatment on land. If not, was the process a sufficient preliminary to the treatment of the sewage on land; and was the land possessed by the Exeter City Council suitable in quantity and quality? The Local Government Board have come to the conclusion that, if the Septic process be adopted, a further treatment of the sewage on land will still be required previous to the effluent being discharged into the river. The Board, however, are not unwilling to accept, by way of experiment, the adoption of the process recommended by the City Surveyor as a method of treatment preliminary to further treatment on land. The Local Government Board will approve of the scheme on the distinct understanding that the City Council, in adopting this system as an experimental substitute for the ordinary process of chemical precipitation, will accept the entire responsibility for the experiment, and will give an undertaking that, in the event of the process failing to give satisfactory results when applied to the sewage of the entire city, they will adopt in its place some process of treating the sewage with chemicals combined with artificial filtration. The Board understand that the tanks can readily be made available for use as part of such a system. With regard to the quantity of land required, the Board would point out that the Exeter City Council must possess themselves

of land for treating the effluent equal to that which is required under schemes in which chemical precipitation and artificial filtration are adopted; that is to say, an acre for each 2000 of the population. The Board will further require the Council to protect themselves from floods, and must therefore stipulate for flood tanks at Duck's Marsh and Belle Isle."

KRYPTON.

ON Monday last, June 6th, 1898, the discovery of yet another element was announced, in a communication made by Prof. Ramsay, of London, to the Academy of Sciences of Paris. The communication was read to the Academy by M. Berthelot. This new element is a gas, and makes a fifth constituent of the atmosphere; it is, however, present in very minute quantities, viz., one part in ten thousand of its volume. Krypton belongs not to the argon, but the helium group; its density is greater than that of nitrogen, being, according to the corrected measurement, 22.47.

The discovery of this new gas is in a way due to the kindness of Dr. Hampson, who supplied Prof. Ramsay with about 750 c.c. of liquid air; this was allowed to evaporate away slowly, until not more than 10 c.c. were left. This gaseous residue was freed from oxygen and nitrogen, and then sparked in the presence of oxygen and caustic soda, when a spectrum was obtained showing the argon lines feebly, but in addition to this a new spectrum was observed.

This spectrum is not yet entirely disentangled from the spectrum of argon; it is, however, characterised by two very brilliant lines, one almost identical with D₃, and another one very strong in the green.

Measurements made with a grating of 14,438 lines to the inch give:—

$$\begin{aligned} D_1 &= 5895.0 \\ D_2 &= 5889.0 \\ D_3 &= 5875.9 \\ D_4 &= 5867.7 \end{aligned}$$

The green line, which is comparable with the helium line in intensity, has the wave-length 5568.8, and the somewhat weaker line which accompanies it has the wave-length 5560.6.

The wave-length of sound was determined in the gas by the method described in the "Argon" paper. The data are—

	I.	II.	III.
Wave-length in air ..	34.17	34.30	34.37
" in gas ..	29.87	30.13	—

Calculating by the formula—

$$\lambda_2 \text{ air} \times \text{density air} : \lambda_2 \text{ gas} \times \text{density gas} :: \gamma \text{ air} : \gamma \text{ gas}$$

$$(34.33)^2 \times 14.479 : (30)^2 \times 22.47 :: 1.408 : 1.666$$

it is seen that, like argon and helium, the new gas is monatomic, and therefore an element.

The atomic weight of krypton will probably be found to be 80.

The Working of Dropping Electrodes.—Wilhelm Palmer.—The apparatus is drawn and described. The mercury is contained in a reservoir connected by a long tube with the capillary funnel, from which it drops through an aqueous solution of calomel on to the other electrode. The experiments are a practical investigation of Nernst's theory of dropping electrodes, and the results fully confirm the theory. The migration of the ions is directed by the contact of a metal and a solution, without breaking the circuit by other than chemical means. The above theory gives, as Nernst has pointed out, a method free from objection of estimating the potential difference between the mercury electrode and the electrolyte.—*Zeit. für Physikalische Chemie*, xxv., No. 2.

THE BRITISH ASSOCIATION.

THE Sixty-eighth Meeting of the British Association for the Advancement of Science will commence in Bristol on Wednesday, the 7th of September, 1898. The following arrangements have been made for the meeting:—

The General Committee will meet on Wednesday, the 7th of September, at 1 p.m., for the election of Sectional Officers, and the despatch of business usually brought before that body. On this occasion the Council will present a Report, giving an account of their proceedings during the past year. The Committee will meet again on Monday, September 12, at 3.15 p.m., for the purpose of appointing Officers for the meeting at Dover in 1899, and of deciding on the place of meeting in 1900. The concluding meeting of this Committee will be held on Wednesday, the 14th of September, at 1 p.m., when the Report of the Committee of Recommendations will be received.

The first General Meeting will be held on Wednesday, the 7th of September, at 8 p.m., when the President will deliver an Address; the Concluding Meeting on Wednesday, the 14th of September, at 2.30 p.m., when the Association will be adjourned to its next place of meeting.

At two Evening Meetings, which will begin at 8.30 p.m., Discourses will be delivered: on the 9th of September by Professor W. J. Sollas, M.A., F.R.S.; and on the 12th of September by Herbert Jackson, Esq.

There will also be other Evening Meetings, at which opportunity will be afforded for general conversation among the members.

Sections.—The following are the titles of the Sections and the names of the members who have been nominated by the Council for the office of President of Section:—

Section.

- | | |
|--|--------------------------------------|
| A. Mathematical and Physical Science | Prof. W. E. AYRTON, F.R.S. |
| B. Chemistry | Prof. F. R. JAPP, Ph.D., F.R.S. |
| C. Geology | W. H. HUDLESTON, Esq., M.A., F.R.S. |
| D. Zoology | Prof. W. F. R. WELDON, F.R.S. |
| E. Geography | Col. GEORGE EARL CHURCH. |
| F. Economic Science and Statistics | JAS. BONAR, Esq., M.A., LL.D. |
| G. Mechanical Science .. | Sir JOHN WOLFE-BARRY, K.C.B., F.R.S. |
| H. Anthropology | E. W. BRABROOK, Esq., C.B., F.S.A. |
| K. Botany | Prof. F. O. BOWER, D.Sc., F.R.S. |

An International Conference on Terrestrial Magnetism and Atmospheric Electricity, which will also meet at Bristol, will be associated with Section A as a Special Department under the presidency of Prof. A. W. RÖCKEL, Sec. R.S.

The different Sections will assemble in the rooms appointed for them, for the reading and discussion of Reports and Papers, on Thursday, September 8; Friday, September 9; Saturday, September 10; Monday, September 12; and Tuesday, September 13.

The acceptance of Papers, and the days on which they are to be read, are determined, as far as possible, by the Organising Committees of the several Sections before the beginning of the Meeting. It is therefore necessary, in order to give an opportunity to the Committees of doing justice to the several Communications, that each Author should prepare an Abstract of his Paper, of a length suitable for insertion in the Annual Report of the Association, and the Council request that he will send it, together with the original Paper, by book-post, on or before August 1, addressed thus—"General Secretaries, British Association,

Burlington House, London, W. For Section.....', Authors who comply with this request, and whose Papers are accepted, will be furnished before the Meeting with printed copies of their Abstracts. If it should be inconvenient to the Author that his Paper should be read on any particular days, he is requested to send information thereof to the Secretaries in a separate note. Abstracts sent in after August 1, but before the end of the Meeting, will appear in the Report, but cannot be printed beforehand.

Reports on the Progress of Science, and of Researches intrusted to Individuals or Committees, must be forwarded to the Secretaries, for presentation to the Organising Committees, accompanied in each case by a statement whether the Author will be present at the Annual Meeting.

No Report, Paper, or Abstract can be inserted in the Report of the Association unless it is in the hands of the Assistant General Secretary before the conclusion of the Meeting.

Tickets.—The Reception Room will be opened on Monday, September 5, at 2 p.m., and on the following days at 8 a.m., for the issue of Tickets to Members, Associates, and Ladies, according to the statement given below, and for supplying Lists and Prices of Lodgings, and other information to strangers on their arrival. No Tickets will be issued after 6 p.m.

Members and Associates.—New Members and Associates can join the Association on the following conditions:—

- I. New Life Members for a composition of £10, which entitles them to receive gratuitously the Annual Reports of the Association which may be published after the date of payment.
- II. New Annual Members for a payment of £3 for the first year. These receive gratuitously the Annual Reports of the Association for the year of admission and for every following year in which they continue to pay a Subscription of £1 without intermission.
- III. Associates for this Meeting only for a payment of £1. They are entitled to receive the Report of this Meeting at two-thirds of the publication price. Associates are not eligible to serve on Committees or to hold any office.

Ladies may become Members or Associates on the same terms as Gentlemen, or they can obtain Ladies' Tickets (transferable to Ladies only) on payment of £1.

E. A. SCHÄFER.
W. C. ROBERTS-AUSTEN, } General Secretaries.
G. GRIFFITH, Assistant General Secretary.

The Preparation of Gentianose.—E. Bourquelot and L. Nardin.—The root of gentian is treated with a hot solution of alcohol at 95°; if the root contains a soluble ferment capable of hydrolysing the gentianose, it must be destroyed. Gentianose exists as perfectly white flakey crystals, which contain no water of crystallisation; it is easily soluble in water, and melts at from 207° to 209°. —*Journ. de Pharm. et de Chim.*, vii., No. 6.

Refraction and Dispersive Power of Silicon in its Compounds.—Gino Abati.—It has already been proved that silicon shows remarkable difference in refractive power, according to the element with which it is combined, but very few compounds have been examined, and the author makes a close investigation of the subject. He used silicon chlorides and bromides, as well as silicon methylate, orthosilicic methylester, propylester, and a solution of silicic hydrate. The refractive indices are taken for lines H_α, H_β, H_γ, and D of the spectrum, and the specific gravities relative to water at 4°. The results obtained show that the value of the refraction constant is not always dependent on the constitution of the compound, but it depends in a much higher degree on the nature of the elements taking part in the compound. —*Zeit. für Physikalische Chemie*, xiv., No. 2.

PROCEEDINGS OF SOCIETIES.

THE ROYAL SOCIETY.

THE Soirée held at the Royal Society's Rooms at Burlington House, on Wednesday evening last, June 8th, was very well attended, the fact of its being a Ladies' night adding much to the enjoyment of the Fellows. The guests were received by Lord Lister and Sir John and Lady Evans.

There were, as usual, a number of interesting exhibits, the apparatus shown by Prof. Roberts-Austen for the Micro-photography of Metals being of great interest.

The Automatic Telephone Exchange System, which proposes to do away with the autocrats of the present Exchanges, was shown by Mr. C. Vautin. We shall be glad when its adoption in London is an accomplished fact.

Two models were shown by Prof. J. Norman Collie, to represent in space the arrangement of the Carbon and Hydrogen Atoms in the Benzene Molecule. In one model the carbon atoms are represented by large, and the hydrogen atoms by small spheres; by rotating the carbon atoms the hydrogen atoms are seen to approach the centre in two sets. In the second model the tetrahedra represent the carbon atoms.

An Interference Dilatometer for the measurement of the thermal expansion of solids, by Fizeau's method of observing the displacement, with rise of temperature, of dark interference bands, was exhibited by Mr. A. Tutton.

Prof. Ramsay showed the spectrum of the new element Krypton; further particulars of this discovery will be found on page 270.

Sir Richard Thorne and Dr. Copeman showed a number of tubes and plates illustrating the Bacteriology of Calf Vaccine Lymph, with special reference to the preparation of Aseptic Glycerinated Lymph; the cultivations and plates prove conclusively that the tubercle bacillus is killed by the glycerin, while the efficiency of the lymph is not affected.

An Improved Heliostat, in which slow motions, enabling the observer to adjust the direction of the reflected beam without taking his eye from the spectroscope (or other instrument), was shown by Dr. Johnstone Stoney. In the adjoining room was the Range-finder, exhibited by Profs. Barr and Stroud: the accuracy of this instrument, under good conditions of working, may be taken as 1 per cent at 3000 yards.

Two demonstrations were given in the Meeting Room—one by Prof. Hele Shaw on the Flow of Water, and the other by Sir Norman Lockyer on the recent Total Solar Eclipse observed at Viziradug.

SOCIÉTÉ D'ENCOURAGEMENT POUR L'INDUSTRIE NATIONALE.

May 19, 1898.

M. CARNOT, President, in the chair.

MM. AUGUSTE and LOUIS LUMIÈRE presented some stereoscopic proofs, printed in colours, obtained by the indirect method. They were obtained by means of three screens, orange, green, and violet, through which three separate photographs were taken. The printing and the superposition of the monochromes were effected by the use of a photographic process with bichromated gums, based on the following fact:—Strong bichromated gum, soluble in the cold, which does not give half-tones when employed alone, acquires this property when insoluble substances are added under certain conditions. If, for example, 5 per cent of bichromate of ammonium and from 5 to 10 per cent of emulsified bromide of silver is

added to a 10 per cent solution of strong gum, and this preparation is spread over a plate of glass, a sensitive surface is produced, which can be exposed under the negative it is desired to reproduce; the plate is then washed with cold water, and a barely visible image is then obtained, formed by the gum which has been made insoluble; this can be coloured as desired. The bromide of silver is removed by hyposulphite. This process gives proofs of all colours, with all the grades of tints present in the negative.

A paper was then presented on behalf of M. CHARPY, on "White, or so-called Anti-friction, Metals."

Followed by a communication by M. Saglio, on "Enamels of High Dilatation."

If the boric acid and the lime in the mineral "pandermite" tend to increase the dilatation of an enamel, they also increase its solubility in acids. The difficulty of the problem lies, therefore, in the union of two properties which appear to be contradictory—high dilatation and insolubility. Trials have, however, proved that the production of enamels with a high dilatation, without the use of lead salts, and with a very satisfactory insolubility, is not impossible. The results of a large number of experiments show that—(1) Silica, kaolin, petalite, and zircon give the enamel infusibility and insolubility, but lower the dilatation. (2) Phosphate of lime increases the dilatation, gives viscosity to the fused enamel, and also gives it a certain amount of insolubility. (3) Cryolite, fluor-spar, and above all rutile, increases the dilatation and the fusibility of the enamel. Phosphoric acid would seem to be particularly interesting, as it appears to combine high dilatation with insolubility.

NOTICES OF BOOKS.

A Manual of Quantitative Chemical Analysis. By E. F. LADD. New York: John Wiley and Sons. London: Chapman and Hall, Lim. 1898. Pp. vi.—82. 12mo.

THE author of this booklet, who is Professor of Chemistry in the North Dakota Agricultural College, and Chemist to the Government Experiment Station, Fargo, North Dakota, explains that it is intended for the use of beginners, and that the methods have been selected to advance the student from the simple analysis to the more complex and difficult, as well as to develop the reasoning power of the student. On this account "explanations of details and reactions have been largely omitted, and the series of questions at the end of each method will test the student's knowledge of chemistry." The course of experimental work covers a good deal of ground:—Gravimetric analysis of metals, acids, and a few simple minerals; volumetric analysis of alkalies and acids; analysis of ashes and soils; analysis of ores of iron, zinc, and lead; electrolysis; examination of sugars, starches, and foods; water analysis; and urine analysis (all within 76 pages).

The author's method of treating the subject will appear from a summary of the first example. Directions are given for dissolving 0.3 gm. pure iron wire in nitric acid, precipitating with ammonium hydrate, filtering, washing, drying, igniting, and weighing as Fe_2O_3 . Then follows an equation (which the author calls a "formula") for calculating the percentage of iron obtained. Three questions succeed:—(1) Why dry ppt. before igniting? (2) Why add HNO_3 ? (3) Is there any danger of loss by volatilisation of the iron? Question No. 2 will doubtless puzzle beginners, and might suggest the answer: "Why not add HNO_3 ?" To dissolve iron in nitric acid without adding the acid is a poser!

The author commends cleanliness and neatness, and recommends "freely using a sponge," which ought to be unnecessary if the student is really neat and avoids spilling liquids on his desk. Better than a sponge would

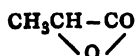
be to cover the desk with a sheet of white paper, and give strict injunctions not to soil it.

The author uses the rules for spelling recommended by the American Association for the Advancement of Science, except where he relapses through forgetfulness, as on p. 29. He uses the objectionable word *titre*, and the fact that he always places it within quotation marks does not abrogate the objection. In the Table of Elements Ramsay appears as "Ramsay" twice, and Hjelm as "Hjehu," but the book is well printed and has an Index. H. C. B.

The Arrangement of Atoms in Space. By J. H. VAN 'T HOFF. Second Edition, Revised and Enlarged. Translated and Edited by ARNOLD EILOART. London, New York, and Bombay: Longmans, Green, and Co. 1898. Pp. 211.

It is twenty-one years since the first edition of this book was published, and the original views with regard to Stereo-chemistry still survive, though the application of them has made great strides during the interval. The possibility of tetrahedral grouping was first suggested to the author by the researches of Pasteur, who had shown the connection between optical activity and crystalline form, and this led to the idea that isomers of opposite rotatory power correspond to an asymmetric grouping and to its mirror-like reflection.

The fundamental idea of Stereo-chemistry is, that in a system of atomic mechanics the molecule will appear as a stable system of material points, and what is dealt with in this volume is nothing else than the spatial—or the real—position of these points, which are the atoms. The asymmetric carbon atom takes first place in the book; and in Chap. II. we have to deal with the division of the inactive mixture, and the temperature of conversion, considered principally with regard to the two tartrates of Pasteur: this is soon followed by a chapter on the position of the radicals in stereomers, the unsaturated carbon compounds and ring formation. With regard to the numerical value of the rotatory power, it must be remembered that this depends on three things—on the wavelength of the light, on the solvent, and on the temperature. The first thing to do is to find the best conditions for obtaining comparable numbers, and here, it is pointed out, it is most essential to take note of the new conception of solution; ring formation appears to have a very extraordinary influence both on the magnitude and the sign of the rotation, the change being first noticed in the case of lactic acid, $[\alpha]_D +2^\circ$ and $+3^\circ$, while the lactone—



has the enormous rotation $[\alpha]_D = -86^\circ$.

After a short chapter on the Stereo-chemistry of Nitrogen Compounds, the book closes with an Appendix by Alfred Werner, on the Stereo-chemical Isomerism of Inorganic Compounds.

The whole book is written in a clear and distinct manner, and will certainly prove to be a valuable help to the comprehension of Stereo-chemistry, and also the work to which the theory has long ago given rise.

Introduction to the Study of Organic Chemistry. By JOHN WADE, B.Sc. (Lond.), London: Swan, Sonnenschein, and Co., Lim. 1898. Pp. 460, and 11 Illustrations.

THE subject of Organic Chemistry is, in the volume now before us, treated experimentally from the beginning, the hypotheses and theories being introduced later on as they are required. There is great advantage in thus dealing with a matter; does not an old saw tell us that "an ounce of practice is worth a pound of theory"? As with engineering, where the 'prentices have to go through the shops as a pre-

liminary to more serious study, so with chemistry, practice should precede theory, though, until quite recently, we are afraid this has not often been the case in view of the always looming written examination.

The book is divided into three principal parts. Part I. Typical compounds of simple construction, subdivided into four sections and nineteen chapters. Beginning with the preparation of pure alcohol by simple distillation, we pass on to the general reactions of alcohol, and the preparation of the ethyl, methyl, and fatty derivatives, the aldehyds, acetone and secondary propyl alcohol, &c.

Part II. is entitled "Aliphatic or Open Chain Compounds," and consists of five sections and nineteen chapters; the first, i.e., Chapter XX., is on the halogen substitution products; then come the hydroxy-substitution products, the glycerol-mesoxalic series, the four lactic acids, &c. Section VIII. deals with the six-carbon derivatives, or sugars; and Section IX. with the aliphatic derivatives of nitrogen, &c.

Part III., the "Aromatic, or Ring Compounds," contains seven sections and twenty-eight chapters. We have first the benzene and phenyl radical, the benzyl-benzoic group,—in fact, all the benzenoid compounds.

It will be noticed from their large number, viz., sixty-seven, that the chapters have purposely been made short; there is very good reason in this, as, the subject being more subdivided, the student is better able to assimilate the ideas than if he had to go on wading through page after page, hardly knowing when to break off.

At the end of nearly every chapter there is a chart, showing the typical formation of the molecules of the bodies just described, and their relations one to another.

There are two appendices. Appendix I., "Laboratory Notes," is full of all kinds of useful instruction, and hints on manipulation in carrying out the various experiments described in the preceding chapters; and Appendix II., on "Qualitative Analysis," in which an excellent scheme is given which will be found useful in determining the nature of artificial mixtures of certain organic compounds, a list of which is given, with inorganic acids, bases, &c., the tests for the individual compounds being given in Appendix I.

There are two indices, a Name Index of six columns and a Subject-matter Index of twenty-nine columns.

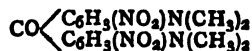
The book should be a valuable addition to those already in use by the University and Technical School Student.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 16, April 18, 1898.

Some Derivatives of Tetramethyl-diamido-benzophenone—E. Grimaux.—To prepare the di-nitrated derivative—



of tetramethyl-diamido-benzophenone, the ketone is dissolved in 20 parts of ordinary sulphuric acid and two molecules of nitrate of potassium added; after standing for twenty-four hours it is poured into water, and the solution saturated with carbonate of sodium. The precipitate is an orange powder and separates out in small crystals from warm water; they resemble bichromate of ammonia in appearance and melt at 165° – 166° . The bromide derivative is prepared by dissolving the ketone in 10 parts of chloroform and adding two molecules of bromine. The chloroform is driven off on the water-bath

and the residue taken up by hydrochloric acid diluted with its own volume of water. The acid solution is precipitated by ammonia, and the white precipitate, after desiccation, is crystallised in 25 parts of boiling alcohol. The di-bromised ketone—



crystallises in hard, brilliant prisms, fusible at 130° — 131° . Both these derivatives have been condensed with dimethylaniline and phenyl- α -naphthylamine by the chloride of phosphorus method and have given new colouring matters.

Influence of Temperature on Chemical Reactions.—Albert Colson.—Though pyrophosphate of silver is not attacked by dry sulphuretted hydrogen at the melting-point of ice, at from 15° to 20° the absorption of SH_2 is noticeable, being equal to 2 m.m. of mercury in twenty-four hours; at 100° it reaches 200 m.m. in one minute with the mean pressure of the gas at about 600 m.m. The ortho-phosphate of zinc, $(\text{PO}_4)_2\text{Zn}_3$, when well dried, behaves in the same way as the silver salts. To determine how the mass of gas absorbed varied at a constant temperature with a diminishing pressure, a certain quantity of SH_2 was put in contact with a large excess of phosphate of zinc at 100° . In fifteen minutes an absorption was noted of—

6.3 c.c. of SH_2 at a mean pressure of 600 m.m.	
4.6 " " " "	430 "
3.2 " " " "	330 "

From this it is seen that the mass of sulphuretted hydrogen decomposed by phosphate of zinc, in a unit of time and at a constant temperature, is proportional to the square of the pressure. The salts of copper are attacked more vigorously than those of silver, but their decomposition does not disengage more heat.

On the Phosphoric Mono-Ethers.—J. Cavalier.—The author finds that monoethylphosphoric acid gives two series of salts, the neutral $\text{PO}_4(\text{C}_2\text{H}_5)_2\text{M}_2$ and the acid $\text{PO}_4(\text{C}_2\text{H}_5)_2\text{MH}$, and that with regard to methyl orange and phthalein they behave like phosphoric acid.

On the Alkaline Sulphantimonites.—M. Pouget.—By acting on the monosulphides of sodium and ammonium with sulphide of antimony, the normal sulphantimonite, SbS_3Na_3 , is obtained; when crystallised it acts powerfully on polarised light. The action of sulphide of ammonium is different to that of sodium and of potassium; the solution is, in fact, only partial, a mass of crystals being formed, while the normal sulphantimonite remains in the mother-liquor.

Zeitschrift für Physikalische Chemie,
Vol. xxv., No. 2.

The Dissociation of Dibasic Organic Acids.—W. A. Smith.—The author measures the relation between the acid strength of the first and second atoms of hydrogen in organic acids. Oswald's theory about the cause of the strength and weakness of the first and second hydrogen atoms is fully confirmed by all the results. In all comparable acids the second hydrogen atom is strong and the first weak in proportion as the hydroxyl groups are nearer to one another or further away. This is proved for acids of the oxalic series, the methyl and ethyl substituted malonic and succinic acids. For the higher homologues of the oxalic acid series the removal of two COOH groups from one another is so great that their mutual influence becomes very small.

Decomposition of some Substances under the Influence of Electric Vibrations.—Alexander von Hemptinne.—The vapour of certain substances is subjected to the action of electric vibrations and the decomposition products examined. The apparatus, of which a drawing is given, is so arranged that the vapour can be introduced at any desired pressure. The substances ex-

amined were methyl alcohol, ethyl alcohol, normal propyl alcohol, isopropyl alcohol, alkyl alcohol, propyl aldehyde, acetone, &c.

New Method of Element Change.—E. Cohen.—The determination of the temperature coefficient of the Latimer Clark cell by Callendar and Barnes shows a break in the curve at 38.75° . The stability curve for ZnSO_4 in water by a similar investigation shows a break at 39.7° . From the dilatometric determination of the density of zinc sulphate solution the author shows that the transformation point, between the stable and unstable phase of the salt, lies at about 38.5° .

Equilibrium in Systems of Three Components, where Two Liquid Phases can Occur.—F. A. H. Schreinmakers.—A mathematical paper unsuitable for abstraction.

Generalisation of the Observation on the Influence of Foreign Bodies on the Transformation Temperature given in the previous Paper.—H. A. Lorentz.

NOTES AND QUERIES.

* Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Ozonator.—A correspondent wishes to know where he can find particulars of the Ozonator of M. Andreoli.

MEETINGS FOR THE WEEK.

WEDNESDAY, 15th.—Microscopical, 8. "Reports on the Foraminifera of the Malsy Archipelago" (continued), by F. W. Millett. Exhibition of Sponges, by E. W. Priest.

THURSDAY, 16th.—Chemical, 8. Ballot for the Election of Fellows. "Preparation of a Standard Acid Solution by Direct Absorption of Hydrogen Chloride," by G. T. Moody, D.Sc. "Researches on the Terpenes—III. Halogen Derivatives of Fenchone and their Reactions," and "IV. On the Oxidation of Fenchone," by J. A. Gardner, M.A., and G. B. Cockburn, B.A.

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Manuals: E. Chr. Hansen, "Practical Studies in Fermentation," London (Spon), 1896. Alfred Jörgensen, "Micro-Organisms and Fermentation," London (F. W. Lyon), 1893.

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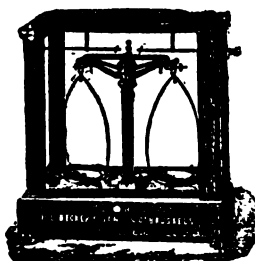
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No. 91. Fig. 11. SHORT BEAM BALANCE for a charge up to 100 grammes in each pan, in french polished glass case, front sliding frame counterpoised, beam divided on both sides in 1-5th part of milligrammes, with new improved arrangement for arrest of pans. Provided with wood stand for taking specific gravity, agate bearings, and sensible to 1-10th part of a milligramme, pans 6 centimetres in diameter, width of bows 9 centimetres. Pans and bows nickel, provided with two rider apparatus, with agate bearings £9 0 0

Fig. 11. No. 91.

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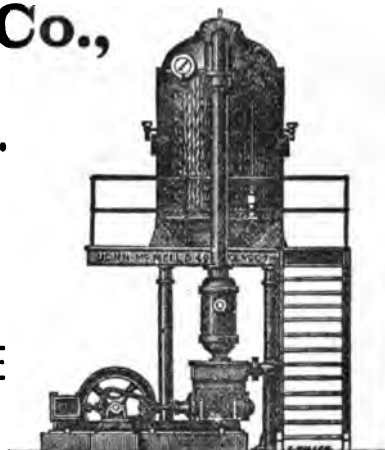
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CONTENTS.

ARTICLES:—	PAGE
On the Lindo-Gladding Method of Determining Potash, by A. L. Winton and H. J. Wheeler.....	275
The Separation and Estimation of Iodine, Chlorine, and Bromine, by Ad. Carnot	276
London Water Supply, by Sir William Crookes, F.R.S., and Prof. Dewar, F.R.S.	278
On the Condition of Oxidation of Manganese Precipitated by the Chlorate Process, by F. A. Gooch and Martha Austin	279
A Determination of the Atomic Weight of Praseodymium and of Neodymium, by Harry C. Jones	280
PROCEEDINGS OF SOCIETIES—	
CHEMICAL SOCIETY—The Boiling-point of Liquid Hydrogen—The Action of Hydrogen Bromide on Carbohydrates—&c. ..	282
PHYSICAL SOCIETY.....	283
CORRESPONDENCE.—Gas and Gases.....	284
NOTICES OF BOOKS.....	284
CHEMICAL NOTICES FROM FOREIGN SOURCES	285
MISCELLANEOUS	285
NOTES AND QUERIES	286
MEETINGS FOR THE WEEK	286

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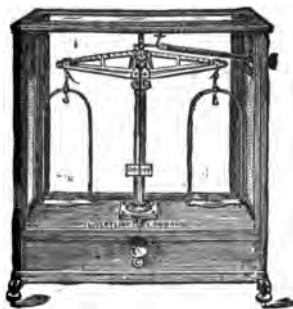
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THE CHEMICAL NEWS.

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ON THE LINDO-GLADDING METHOD OF DETERMINING POTASH.

By A. L. WINTON,
Chemist, Conn. Agricultural Experiment Station, and Reporter on
Methods of Potash Determination for the Association of Official
Agricultural Chemists for the Years 1896 and 1897;

and
H. J. WHEELER,
Chemist, R. I. Agricultural Experiment Station, and Reporter on
Methods of Potash Determination for the Association of Official
Agricultural Chemists for the Years 1894 and 1895.

(Concluded from p. 265.)

BREYER and Schweitzer state that it is very "doubtful whether the method of Lindo-Gladding is a simplification of Fresenius's method." At all events, it should be stated that the members of the Association, after having given both methods an extended trial, find the latter decidedly shorter.

Breyer and Schweitzer state that potassium platinichloride obtained by them by the Lindo-Gladding method contained impurities, notably magnesium and calcium sulphates, but as they give no figures it is to be assumed that the amounts present were not determinable.

If the potash exists as chloride, and chlorides of sodium, magnesium, and calcium are the only soluble impurities, evaporation with platinum solution and washing with alcohol should yield pure potassium platinichloride, the salts of chloroplatinic acid, with the other bases, being readily soluble in alcohol. Were this not true neither the Lindo-Gladding nor the Stassfurt method would be practicable.

If, however, the bases mentioned are combined wholly or in part with sulphuric acid, the potassium platinichloride, obtained by the Lindo-Gladding method is usually, after the first washing, contaminated with magnesium and calcium sulphates; subsequent washing with ammonium chloride solution being depended upon to remove these impurities.

Magnesium sulphate is readily soluble in the ammonium chloride solution. Even when a very considerable amount is present, the first addition of the reagent removes almost instantly the greater part of it. Calcium sulphate does not dissolve so readily, but it is contained only in small amount in the potash manure salts on sale in the United States, and the treatment with ammonium chloride solution effectually removes both this salt and the magnesium sulphate.

That this is true is shown by the figures obtained by one of us (A. L. W.), which are given in Table III. The results, calculated from the weights of K_2PtCl_6 (together with many others, equally accurate, obtained by seven American chemists), were reported to the Association of Official Agricultural Chemists in 1896 (U.S. Department of Agriculture, Div. of Chemistry, *Bull.* 49, pp. 27-38), but those calculated from the weights of metallic platinum are here given for the first time.

The materials used were chemically pure potassium sulphate, which had been ignited until all water was removed, and three mixtures of salts representing the water-soluble ingredients, other than potash salt, contained in sulphate of potash (90 per cent), sulphate of potash and magnesia, and kainit. The solutions for analysis, representing half-grm. portions of the commercial potash salts named, were prepared by mixing weighed portions of the pure salt with the corresponding amounts of the mixtures of impurities, dissolved in each case in 25 c.c. of water. The determination of potash in these solutions presented

the same analytical difficulties as would have been encountered in the analysis of the real commercial salts, but the exact amount of potash present was in each case known, and the results obtained could be compared with theory.

The Lindo-Gladding method of the Association (U.S. Dept. of Agric., Div. of Chem., *Bull.* 46, p. 23) was strictly followed, except in the case of the kainit solution, which was evaporated directly with platinum solution, without previous separation of lime, as was originally directed by Gladding (U.S. Dept. of Agric., Div. of Chem., *Bull.* 7, p. 41). The potassium platinichloride was weighed on Gooch crucibles, reduced in hydrogen, washed with hot water, ignited, and weighed as metallic platinum; all of which was accomplished without transfer. The small amounts of potash in the impurities were determined by careful analysis and calculated as potassium sulphate.

The conversion factors employed were:— K_2PtCl_6 to K_2SO_4 , 0.3587; and Pt to K_2SO_4 , 0.8937. These were derived from the following atomic weights:—Pt=195.0, K=39.11, Cl=35.45, S=32.06, O=16.0.

The factors which have commonly been used in potash determinations (0.3056 for converting K_2PtCl_6 to KCl, 0.3570 to K_2SO_4 , and 0.19308 to K_2O) are those based on an atomic weight of platinum long since abandoned. Fresenius (*Zeit. Anal. Chem.*, xxi., 238) found in some determinations made on pure potassium chloride shortly after the now-accepted atomic weight of platinum was determined by Seubert (*Ann. Chem.*, ccvii., p. 1), that the factor 0.3056 gave results nearer theory than the factor derived from the later atomic weights. But it has been shown by one of us (A. L. W., *Journ. Am. Chem. Soc.*, xvii., p. 453) that when platinum solution is added to a concentrated solution of the potash salt, as was done by Fresenius, much of the potassium platinichloride separates out at once as a finely-divided precipitate, which, examined under the microscope, may be seen to consist of radiating crystals, enclosing numerous globular cavities containing liquid. Part of the enclosed liquid is slowly driven off at 100°, more at 130°, and still more at 160°C. As Fresenius dried at 130°, the water was presumably only partially removed, and for this reason the lower factor gave the best results. Winton has further shown, however, that when the solution of the potash salt is so dilute that the addition of the platinum solution either forms no immediate precipitate, or one that dissolves on heating, the potassium platinichloride deposits slowly on evaporation in the form of coarsely granular crystals, which appear under the microscope to be almost entirely free from enclosed liquid, and which dry as completely on heating three hours at 100° as when heated for many hours at 130° and 160°C. When the salt is obtained by this latter method, as was the case in all the determinations of Tables I., II., and III., the factors based on the latest atomic weights give satisfactory results. The fact that the results (given in Table III.) obtained by the Lindo-Gladding method, and calculated by the revised factor, agree closely with those calculated from the weight of the platinum and with theory, is of itself a strong indication of the purity of the potassium platinichloride and the perfection of the process. It is only in the case of methods which yield an impure double salt that it is necessary to resort to the unscientific procedure of retaining incorrect factors in order to compensate for errors of the method.

Vogel and Haefke (*Die Landw. Versuchs-Stationen*, 1896, xlvii., pp. 115-116), as their first point against the Lindo-Gladding method, claim that the addition of sodium chloride is unnecessary, and is liable to prove a source of error. This intended criticism was made in 1896, five years after the matter was first considered by the Association, and three years after the Association had ceased to employ it—a fact which they might have ascertained had they consulted the published records of the Association.

TABLE III.—Results by the Lindo-Gladding Method on Mixtures representing Sulphate of Potash (90 per Cent), Sulphate of Potash and Magnesia, and Kainit.

Taken. Impurities corresponding to 0.5 grm. of—	Taken. K ₂ SO ₄ pure salt.	Taken. K ₂ SO ₄ in impurities.	Taken. K ₂ SO ₄ total.	Found. K ₂ PtCl ₆ .	Found. Pt.	Results calculated from weight of K ₂ PtCl ₆ .		Results calculated from weight of Pt.	
						Found. K ₂ SO ₄ Grm.	Error. K ₂ SO ₄ Grm.	Found. K ₂ SO ₄ Grm.	Error. K ₂ SO ₄ Grm.
Sulphate of potash (90 per cent) ..	0.4042	0.0002	0.4044	1.1248	0.4523	0.4035	-0.0009	0.4042	-0.0002
	0.4121	0.0002	0.4123	1.1483	0.4615	0.4119	-0.0004	0.4124	+0.0001
	0.4110	0.0002	0.4112	1.1448	0.4591	0.4106	-0.0006	0.4103	-0.0009
Sulphate of potash and magnesia ..	0.3040	0.0003	0.3043	0.8470	0.3408	0.3038	-0.0005	0.3046	+0.0003
	0.2686	0.0003	0.2689	0.7490	0.3017	0.2687	-0.0002	0.2696	+0.0007
	0.2566	0.0003	0.2569	0.7136	0.2866	0.2560	-0.0009	0.2561	-0.0006
Kainit	0.1387	0.0005	0.1392	0.3860	0.1547	0.1385	-0.0007	0.1383	-0.0003
	0.1727	0.0005	0.1732	0.4807	0.1922	0.1724	-0.0008	0.1718	-0.0004

Again, Vogel and Haefke, after calling attention to the well-known fact of the double decomposition which results when potassium platinichloride is acted upon by ammonium chloride, further add that proof is lacking to show that the ammonium chloride, even after having been brought in contact with potassium platinichloride, is not capable of effecting still further double decomposition. Assuming that such further action results to a degree which practically interferes with the accuracy of the method, they find therein another point against it. In fact, two years before this theoretical criticism was published, the Association had shown that the wash solution was capable, under greatly exaggerated conditions of treatment, of further effecting a considerable amount of double decomposition; but at the same time, as has already been shown, it was proved that in actual analytical work the amount of error thus introduced was practically so small as to furnish no valid objection to the method. Vogel and Haefke could have learned these facts also before their criticisms appeared had they consulted the published records of the Association whose official methods they were discussing. It must be conceded that, upon theoretical grounds only, many valuable and reliable methods of analysis would have to be discarded if the solvent action of the wash liquids, regardless of the degree of solubility, were the sole basis for such a procedure.

The variation in the time employed in washing the potassium platinichloride, due to unequal rapidity of filtration and the size of the crystals to be washed, are also urged by Vogel and Haefke as logical objections to the method. However, by the use of the Gooch crucible, and by precipitating the potassium platinichloride from dilute solutions (by which large crystals are obtained), it must be obvious—and, in fact, it has been shown by abundant analytical data—that these objections hold only from a theoretical, and not from a practical standpoint.

Vogel and Haefke still further assert, based upon assumption or upon the previously mentioned statements of Breyer and Schweitzer, that the potassium platinichloride obtained by the Lindo-Gladding method contains many impurities, and therefore that it is not advisable to weigh the double salt as such. However, as has been shown by the results in Table III., the potassium platinichloride, obtained by the Lindo-Gladding method, even in the presence of such impurities as are in sulphate of potash (90 per cent) and the commercial sulphate of potash and magnesia, as well as in kainit, is practically pure, and the results accord as closely as could be desired with those obtained by reduction and weighing the metallic platinum.

From what has been said above, it will be seen that Vogel and Haefke were not well informed as to the work of the Association, nor in relation to the status of the method in vogue when their unfavourable criticisms were made; furthermore, they present no analytical data of their

own in support of their assumptions, and, in the main, the points which they sought to make were but a reiteration of the previous unfavourable criticisms of Breyer and Schweitzer, which have been shown by abundant data to have been, so far as practical considerations go, utterly unfounded.

THE SEPARATION AND ESTIMATION OF IODINE, CHLORINE, AND BROMINE.

By AD. CARNOT.

MANY methods have already been proposed for the estimation of these three bodies in a mixture of haloid salts. By offering still another one, I am actuated by the fact that it appears to have the qualities of simplicity, rapidity, and exactness, which cannot be found in any of the already known methods. I have used the excellent advice given by Fresenius for the estimation of the iodine, and I also took advantage of some observations made by M. Dechaux and M. Baubigny relative to bromine.

The method is founded on the following reactions. In a mixture of chlorides, bromides, and iodides in solution, sulphuric acid charged with nitrous vapours entirely displaces iodine in the cold, without acting on hydrochloric or hydrobromic acid in any manner whatever; the iodine can thus be entirely dissolved and removed by sulphide of carbon. On adding sulphuric and chromic acids the bromine is isolated very slowly in the cold, but by heating up to about 100° for from half an hour to an hour, it can be entirely isolated, by dissolving in sulphide of carbon on cooling; no trace of chlorine is set at liberty in one case or the other. The estimation of this body can then be made by means of nitrate of silver.

The free iodine is estimated volumetrically by hyposulphite of sodium, added to the sulphide of carbon until exact decolouration takes place; for bromine the operation is similar, but iodide of potassium must be first added, after which the violet colour given by the free iodine must be exactly neutralised by means of titrated hyposulphite of sodium.

A series of experiments carried out on the most varied proportions of chloride, bromide, and iodide have proved the trustworthiness of the results when the necessary precautions are taken.

1. *Iodine*.—The neutral solution of the salts, diluted to about 200 cubic centimetres, is placed in a spherical funnel of from 350 to 400 c.c. capacity, well plugged at the upper part by a ground stopper, and at the lower extremity by a glass tap made of sufficiently thin glass that there will be no risk of breakage on heating. Into the cold solution ten drops of sulphuric acid saturated with nitrous vapours (produced by the reaction of concentrated nitric acid on starch) are poured, after which 10 to 15 c.c.

Proportions used in the experiments.						Hypo sulphite used for the—			Element found.		
Iodide. M.grms.	Bromide. M.grms.	Chloride. M.grms.	I. M.grms.	Br. M.grms.	Cl. M.grms.	I. C.c.	Br. C.c.	AgCl weighed. M.grms.	I. M.grms.	Br. M.grms.	Cl. M.grms.
200	100	200	153.0	67.2	95.2	22.3	15.2	380	152.5	65.3	94.0
100	100	100	76.5	67.2	47.6	11.2	15.4	—	76.6	66.2	—
100	200	100	76.5	134.4	47.6	11.2	31.0	192	76.6	133.3	47.5
20	40	200	15.3	26.9	95.2	2.2	6.3	—	15.0	27.1	—
5	50	200	3.8	33.6	95.2	0.6	7.8	—	4.1	33.5	—
1	100	600	0.8	67.2	285.6	0.1	15.3	—	0.7	65.8	—
—	20	500	—	13.4	238.0	—	3.1	—	—	13.3	—
—	10	200	—	6.7	95.2	—	1.5	—	—	6.4	—
—	5	200	—	3.4	95.2	—	0.7	—	—	3.0	—
—	1	200	—	0.7	95.2	—	0.1	—	—	0.4	—
100	100	20	76.5	67.2	9.5	11.2	15.5	39	76.6	66.6	9.6
100	100	5	76.5	67.2	2.4	11.2	15.6	10	76.6	67.1	2.5

of pure sulphide of carbon is introduced. The glass stopper is firmly fixed and the apparatus thoroughly shaken several times; the sulphide of carbon is then allowed to collect, while the bulb is carefully rotated to collect the small drops of sulphide which adhere to the surface of the glass. If there is much iodine present the sulphide of carbon will now be of a deep violet colour, or if, on the other hand, there is only a small quantity, the colour will be of a pale rose tint; this is easily seen in the aqueous solution, and it also fills the lower part of the bulb and the fine tube down to the stopcock.

The stopcock is then carefully turned, and the sulphide of carbon run on to a filter previously moistened with water; the tap must be quickly turned off the moment the aqueous solution is about to pass. Another 10 c.c. of sulphide of carbon is then added and the whole shaken as before; as a rule there is now very little colouration by iodine; four or five drops of nitrous sulphuric acid are again added, and after further agitation there should be no change of colour if the first operation has been properly carried out. The solution is now allowed to settle again, and the sulphide is collected on the same filter, which must be covered by a clock-glass to prevent evaporation.

The addition of two or three cubic centimetres of sulphide of carbon and one or two drops of nitrous sulphuric acid will serve to collect the small droplets which still remain on the surface of the solution; this also assures one of the complete displacement of the iodine, besides serving to wash out the small quantity of faintly coloured sulphide still remaining in the stopcock.

The sulphide of carbon which has been collected on the moistened filter must then be well washed with cold water. The first washings only are collected and added to the aqueous solution in the glass bulb for the purpose of analysis. By piercing the filter the sulphide of carbon can be run into a small ground-stoppered bottle, in which is about 30 c.c. of a half per cent of bicarbonate of soda. To this mixture is then added, by means of a graduated burette, a titrated solution of hyposulphite of sodium (either decinormal or centinormal) until the complete decolouration of the sulphide of carbon. The solution must be well shaken after each addition of the reducing agent. The reaction is very distinct, and the results are as accurate as possible, not only in the presence of chlorides, as was observed by Fresenius, but also in the presence of bromides.

2. *Bromine*.—For the estimation of bromine a few c.c. of chromic acid at 10 per cent and three or four c.c. of sulphuric acid diluted with its own volume of water are added to the contents of the funnel, which must now be firmly closed and placed in a hot water bath for at least an hour. It is then allowed to cool completely, after which sulphide of carbon is added and the operation completed in the same manner as has just been described in the case of iodine, by three successive exhaustions. The sulphide of carbon is then received on a moistened filter, and washed with cold water until the latter is no longer acid.

The sulphide is then run into a ground glass stoppered

flask, to which a small quantity of iodide of potassium and about 30 c.c. of bicarbonate of sodium is added. The flask must now be well shaken several times. The bromine displaces an equivalent quantity of iodine, which, when set free, gives on dissolution a violet colour to the liquid, of a much stronger character than the yellowish brown tint caused by the original bromine. The estimation of the iodine is made as before, by titrating with hyposulphite of sodium, and to arrive at the corresponding weight of bromine we have simply to multiply the weight of iodine by 80/127.

3. *Chlorine*.—The acid solution, from which the iodine and bromine has been removed, and to which the first wash-waters have been added, must now be poured into a beaker and diluted with about 500 c.c. of water; nitrate of silver is then added and the solution heated to collect the chloride. The precipitate will be slightly coloured by the presence of a small quantity of chromate of silver; to purify it the filtrate must be decanted after cooling, and the volume made up by warm water slightly acidulated with nitric acid; this is once more allowed to cool and is then washed by decantation. The chloride of silver, which should now be perfectly white, is collected on a tared filter, dried, and weighed with the usual precautions.

This estimation of the chlorine is, of the three, the one to which, as a rule, the least importance may be attached. As a matter of fact, instead of estimating directly, as I have just described, it would generally be quite sufficient to estimate by difference; this can easily and rapidly be done volumetrically in the following manner. The determination should be carried out on the solution, from which the iodine has already been removed by the first operation. This must be divided into two portions; the first is only used for the estimation of the bromine, as has just been described; the other is precipitated by a measured quantity of titrated nitrate of silver; the excess of silver is then estimated by means of sulphocyanide, using ferric iron as indicator; according to the quantity of the bromine found the amount of nitrate of silver used is known, and thus the chlorine is calculated.

We can also, especially if the chlorides are present in much greater quantity than the bromides and iodides, take only 1/10th or 1/50th of the original neutral solution, add chromate of potassium to it as indicator, and then, by means of a graduated burette, run in titrated nitrate of silver until the red colour of the chromate appears. By deducting from the nitrate of silver used the amount which corresponds to the iodine and to the bromine, we get the quantity which was precipitated by the chlorine.

The chlorine can thus be very easily estimated by either of these methods.

The accompanying table, showing the results of a series of trials carried out on either very large or very small proportions of each of these three bodies, enables us to appreciate the accuracy which can generally be attained by this method.

For a simple qualitative research for small quantities of iodides or bromides in the presence of a large excess of

an alkaline chloride, we recommend the following manner of working:—

1. Separate the iodine in a small quantity of the neutral solution by nitrous-sulphuric acid, and collect it in a few drops of sulphide of carbon. The violet or rose colouration is extremely sensitive.

2. The iodine having been eliminated, add to the liquid, placed in a small flask, a little chromic and sulphuric acids; then heat to boiling, while keeping the flask covered with yellow fluoresceine paper, as described by M. Baubigny, who pointed out its sensitiveness to bromine. The very smallest quantities of bromine will be revealed by the characteristic rose-colour. — *Bull. Soc. Chim.*, Series 3, vol. xix.-xx., No. 6.

LONDON WATER SUPPLY.

REPORT ON THE COMPOSITION AND QUALITY OF DAILY SAMPLES OF THE WATER SUPPLIED TO LONDON FOR THE MONTH ENDING MAY 31ST, 1898.

By SIR WILLIAM CROOKES, F.R.S.,

and

PROFESSOR DEWAR, F.R.S.

To MAJOR-GENERAL A. DE COURCY SCOTT, R.E.,
Water Examiner, Metropolis Water Act, 1871.

London, June 10th, 1898.

SIR,—We submit herewith, at the request of the Directors, the results of our analyses of the 175 samples of water collected by us during the past month, at the several places and on the several days indicated, from the mains of the London Water Companies taking their supply from the Thames and Lea.

In Table I. we have recorded the analyses in detail of samples, one taken daily, from May 2nd to May 31st inclusive. The purity of the water, in respect to organic matter, has been determined by the Oxygen and Combustion processes; and the results of our analyses by these methods are stated in Columns XIV. to XVIII.

We have recorded in Table II. the tint of the several samples of water, as determined by the colour-meter described in previous reports.

In Table III. we have recorded the oxygen required to oxidise the organic matter in all the samples submitted to analysis.

Of the 175 samples examined by us, all were found to be clear, bright, and well filtered.

For the first time this year we have to record an excess of rain at Oxford. The actual fall during the month of May was 2.33 inches, the average fall during the last thirty years is 1.83 inches, we thus have an excess for the month of 0.5 inch; this reduces the deficiency for the year to 2.57 inches.

Our bacteriological examinations of 241 samples have given the results recorded in the following table; we have also examined 41 other samples, from special wells, stand-pipes, &c., making a total of 282 samples in all:—

	Microbes per c.c.
New River, unfiltered (mean of 24 samples) ..	227
New River, filtered (mean of 24 samples) ..	8
Thames, unfiltered (mean of 25 samples) ..	1473
Thames water, from the clear water wells of five Thames-derived supplies (mean of 120 samples)	23
Ditto ditto highest	578
Ditto ditto lowest	0
River Lea, unfiltered (mean of 24 samples) ..	7693
River Lea, from the East London Company's clear water well (mean of 24 samples) ..	25

On the 16th of the month one sample was found to give abnormally bad results. The affected Company's Engineer was immediately telephoned to, and we were informed

that a defect had been found in one of the filters, and this defect was being rectified when the Engineer received our message. The quality of the water was soon improved, and has since remained good.

The increase of rainfall has made itself apparent on our results, both chemical and bacteriological, but only to a slight extent; the water remaining in a very satisfactory condition.

We are, Sir,

Your obedient Servants,
WILLIAM CROOKES.
JAMES DEWAR.

ON THE CONDITION OF OXIDATION OF MANGANESE PRECIPITATED BY THE CHLORATE PROCESS.*

By F. A. GOOCH and MARTHA AUSTIN.

(Concluded from p. 270).

CONFINING our attention to the last two simpler iodometric methods—the reduction of the higher oxide by an acidified iodide on the one hand and by arsenious acid on the other—we made, first, some experiments to determine the accuracy with which manganese may be thus estimated. We used for the manganese compound of known oxidising power a solution of potassium permanganate filtered carefully through asbestos and standardised against ammonium oxalate which had been found to be the exact equivalent of a specially-prepared lead oxalate. For each experiment a definite portion of this solution was drawn from a burette and treated with a solution of pure manganous sulphate until the colour of the permanganate had vanished, thus precipitating a hydrous oxide approximating quite closely probably to the condition of oxidation of the dioxide, but containing at all events, whatever its actual composition might be, the exact amount of available oxygen originally in the permanganate. In the experiments of the following table this precipitate was treated with a solution of potassium iodide (6 grms.) and tartaric acid (20 grms.), by which the freshly-prepared hydrate is dissolved quite as well as by the iodide and hydrochloric acid of Pickering's original method and with less risk of evolution of iodine outside the main reaction. From the iodine found by titration with this sulphate we have calculated the weight of manganese dioxide which would liberate it; and a comparison of this value with the amount of the dioxide theoretically precipitated by the interaction of the known permanganate and the sulphate, upon the assumption that two molecules of the former throw down five molecules of the hydrated dioxide, should disclose the error of the analytical process when applied to the estimation of manganese dioxide. In all probability the assumption that it is the dioxide that is precipitated, and which afterwards acts upon the iodide, is not quite true under the conditions, since the precipitation takes place in presence of an excess of a manganous salt; but for our purpose it does not matter, since we are in effect simply dealing with the oxidising power of a known amount of permanganate.

Thus, it is obvious that the mean error of the results is practically inconsiderable, varying between extremes of -0.0004 grm. and $+0.0007$ on 0.1351 grm. of manganese dioxide.

In the experiments of Table III. the precipitated oxide was treated with an excess of a standard arsenious acid solution and 5 c.m.* of sulphuric acid of half strength, and the whole was heated until the manganese dioxide was dissolved. To this liquid was added tartaric acid (20 grms.) to prevent the precipitation of the manganese and

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science* vol. v., p. 260.

the oxidation by iodine in the subsequent titration, the acid was neutralised by acid potassium carbonate, and the arsenic still remaining in the arsenious condition titrated by standard iodine.

TABLE II.

Mn theoretically precipitated by KMnO_4 as MnO_2 . Grm.	Mn in MnO_2 corresponding to iodine found. Grm.	Error of the analytical process (applied to MnO_2) in terms of Mn. Grm.
0'1351	0'1347	0'0004—
0'1351	0'1347	0'0004—
0'1351	0'1350	0'0001—
0'1351	0'1353	0'0002+
0'1351	0'1358	0'0007+
0'1351	0'1353	0'0002+

TABLE III.

Mn precipitated by action of KMnO_4 on MnSO_4 as MnO_2 . Grm.	Mn in MnO_2 corresponding to As_2O_3 oxidised. Grm.	Error of the process in terms of Mn. Grm.
0'1392	0'1396	0'0004—
0'1109	0'1117	0'0008—
0'1112	0'1117	0'0005—
0'1109	0'1117	0'0008—
0'1109	0'1117	0'0008—
0'1117	0'1125	0'0008—

It is clear that either of these methods of reduction, the action of an acidified iodide or that of arsenious acid, is capable of yielding fairly accurate indications when we have to deal with a pure salt of manganese. When, however, the manganese is associated with a considerable amount of iron, as is frequently the case, it becomes a matter of necessity to separate the manganese before attempting its estimation. For this purpose, the "chlorate process" is by far the simplest of those generally used, and though it has been the subject of much discussion it is at present the method of separation most widely used by the practical chemists, whether the final estimation of the manganese is made gravimetrically, as in Ford's process, or volumetrically, as in the methods of Volhard, Williams, or Pattinson.

Definite portions of the solution of pure manganous chloride were drawn from a burette into an Erlenmeyer flask of 300 c.c. capacity, evaporated to dryness, precipitated by the "chlorate process" with the modifications given in detail above. The oxide, after careful washing, was returned with the asbestos to the flask, and treated by one or other of the methods to be described. It was either treated with potassium iodide (5 grms.) and sulphuric acid (10 c.c.) of half strength, the iodine set free being estimated by thiosulphate; or it was heated with an excess of standard arsenious acid and 20 c.c. of sulphuric acid of half strength, and, after cooling, adding 5 grms. of the Rochelle salt and neutralising with acid potassium carbonate, the arsenious acid remaining unoxidised was estimated with standard iodine. In Table IV. are given the results obtained from this work.

The results show plainly that, while the manganese is so completely precipitated in the chlorate process of oxidation when properly conducted that only insignificant traces may escape, the condition of oxidation cannot be taken to be that of the dioxide. The average error thus put upon the determination of the manganese known to be present is more than 2 per cent. It follows, as a matter of course, that the indications of any process which rests upon the assumption that the oxygen value of the manganese compound precipitated in the chlorate process corresponds to that of the dioxide must of necessity be erroneous. If, therefore, the chlorate method is to be employed for the separation of the manganese, it is obvious that precautions must be taken to secure a definite condition of oxidation of the manganese before processes which depend upon the oxygen value of the higher oxide may be applied for the estimation of that element. The process which in our hands seems to give the oxide in

TABLE IV.

Mn taken in the form of manganous chloride. Grm.	Mn found upon the hypothesis that MnO_2 is precipitated. Grm.	Error. Grm.	Mn found in the filtrate after evaporation and treatment with bromine and ammonia. Grm.
<i>By Reduction with Potassium Iodide.</i>			
0'1225	0'1183	0'0042—	0'00006
0'1225	0'1177	0'0048—	Trace
0'1225	0'1180	0'0045—	0'00008
0'1225	0'1169	0'0056—	Trace
<i>By Reduction with Arsenious Acid.</i>			
0'1222	0'1189	0'0033—	Not determined.
0'1222	0'1191	0'0031—	" "
0'1222	0'1199	0'0023—	" "
0'1222	0'1200	0'0022—	" "
0'1222	0'1186	0'0036—	None
0'1222	0'1187	0'0035—	0'0001
0'1222	0'1189	0'0033—	0'0002
0'1222	0'1194	0'0028—	Trace
0'1222	0'1205	0'0017—	0'0001

definite condition is based upon the observations of Wright and Menke (*Journ. Chem. Soc.*, xxxvii., 36) that a dilute solution of potassium permanganate acting in excess, at 80° C., in the presence of zinc sulphate, and in thorough mixture upon manganous sulphate, yields an oxide which, though combined with alkali, holds the oxygen exactly in the proportion corresponding to the dioxide. Three-fifths of the manganese in such a precipitate represents the amount of that element originally present in the manganous salt. In the following table are given the results of experiments in which manganese was determined iodometrically after the interpolation of the permanganate treatment.

In these experiments a solution of manganous chloride of known strength was drawn from a burette, evaporated to dryness in a small beaker, heated with nitric acid until there was no evidence of the presence of nitrogen oxides. Strong nitric acid was poured in until the volume was 85 c.m.³, sodium chlorate (5 grms.) was added carefully, the liquid was boiled five minutes, more nitric acid (15 c.m.³) and a few crystals of the chlorate were introduced, and the solution brought to boiling temperature again. After cooling, the liquid was filtered on asbestos and washed with water, and the oxide upon the asbestos and walls of the beaker was dissolved in 2 c.m.³ of hydrochloric acid. After diluting a little, the solution was evaporated with 5 c.m.³ of strong sulphuric acid until no more hydrochloric acid remained. The solution of manganous sulphate (not exceeding 0.5 gm. of the salt), very nearly neutralised by potassium carbonate, was mixed with a solution of zinc sulphate (2 grms.) and a freshly and carefully filtered dilute solution of potassium permanganate (1.5 gm. of the salt); the liquid, amounting now to about 500 c.m.³, was heated to 80° C. and acid potassium carbonate added, in quantity a little more than enough to neutralise the remnant of the acid present. The precipitate was collected upon asbestos, and after careful washing was returned to the flask in which the precipitation had been made. The oxygen value of the oxide was determined by one or other of the methods described. In the one case the flask was fitted with a paraffined stopper having two bores, one holding a Will and Varrentrap absorption apparatus (in which a solution of potassium iodide dissolved any escaping iodine), the other a small separating funnel. Sulphuric acid and potassium iodide in solution were run in through the funnel, the iodine set free was titrated with thiosulphate—the amount of manganese being reckoned from the iodine set free. The results of this work follow in the first part of Table V. In the second case the dioxide obtained in the manner described above was reduced by warming gently with a decinormal solution of arsenious acid. After cooling, and neutralising with acid potassium carbonate in the presence

of Rochelle salt, the excess of the arsenious acid was estimated with iodine in the presence of starch. The estimation by this method gave the results recorded in the second part of Table V.

TABLE V.

Mn taken in the form of chloride. Grm.	Mn found upon the hypothesis that MnO ₂ is the oxide finally obtained. Grm.	Error. Grm.
<i>By Reduction with Potassium Iodide.</i>		
0.0643	0.0637	0.0006—
0.0643	0.0642	0.0001—
0.0643	0.0642	0.0001—
0.0651	0.0651	0.0000
0.1125	0.1121	0.0004—
0.1125	0.1121	0.0004—
0.1125	0.1120	0.0005—
0.1214	0.1206	0.0008—
0.1214	0.1207	0.0007—
0.1214	0.1223	0.0009+
0.1214	0.1214	0.0000
<i>By Reduction with Arsenious Oxide.</i>		
0.1213	0.1212	0.0001—
0.1213	0.1201	0.0012—
0.1213	0.1203	0.0010—
0.1213	0.1208	0.0005—

These results show plainly that if the precautions to which attention has been directed are taken, viz., dilution of the solution and heating to 80° C., presence of zinc sulphate, and (most essential of all) the almost complete neutralisation of free acid before the addition of the potassium permanganate, the manganese dioxide precipitated by the chlorate process from pure manganous nitrate may subsequently, after reduction, be brought by the permanganate treatment so nearly to the full degree of oxidation represented by the symbol MnO₂, that the amount of manganese originally treated may be calculated with a very fair degree of accuracy from the oxygen value of three-fifths of the oxide found. We do not recommend this procedure as a rapid analytical method; our purpose is accomplished when the fact is brought plainly to view that the oxide precipitated by the chlorate process is not the dioxide, but that it may be made such by subsequent treatment.

A DETERMINATION OF THE ATOMIC WEIGHT OF PRASEODYMIUM AND OF NEODYMIUM.*

By HARRY C. JONES.

THE determinations of the atomic weight of the supposed element didymium have been discussed, to a greater or less extent, by Meyer and Seubert ("Atomgewichte," 218), Ostwald ("Lehrb., Allgem. Chem.," i., 70), and Clarke ("A Re-calculation of the Atomic Weights," 351—353). Yet, for the sake of completeness and reference, a brief account of what has been done will be given here.

Marignac (*Ann. Chim. Phys.* [3], xxvii., 231; xxxviii., 151) precipitated the sulphuric acid from a solution of didymium sulphate by means of barium chloride, but found that when the precipitate was boiled, in the presence of even a large excess of the chloride, it still contained didymium sulphate. His best results were obtained by precipitating the didymium from a solution of the sulphate, as oxalate, by means of oxalic acid, and decomposing the oxalate at a very high temperature, to avoid the presence of superoxide. He found, as the mean of

five determinations, 143.58 (SO₃=80) as the atomic weight of didymium.

Hermann (*J. Prakt. Chem.*, lxxxii., 387) precipitated the didymium from a solution of the sulphate, as oxalate, by means of ammonium oxalate, decomposed the oxalate by heat, and weighed the oxide. He found 146.7 for the atomic weight of the element. The determination of the chlorine in the chloride gave 142.2 as the atomic weight of didymium. Zschiesche (*J. Prakt. Chem.*, cvii., 75) decomposed the sulphate to the oxide, and obtained the value 141.8. Erk (*Ztschr. Anal. Chem.*, x., 509), using the method of Hermann, found the atomic weight of didymium to be 142.35. He also precipitated the sulphuric acid from the solution, out of which the didymium had been separated by oxalic acid, as barium sulphate. By this method the atomic weight was found to be 142.29. He employed, in addition, the method which Marignac first used, precipitating the sulphuric acid directly from a hydrochloric acid solution of didymium sulphate, as barium sulphate, and obtained the figure 143.46. This is necessarily erroneous, since, as Marignac has pointed out, some didymium sulphate is always carried down under these conditions.

Hillebrand (*Ann. der Phys. Pogg.*, clviii., 71) converted a weighed amount of the metal into the nitrate, and then into the oxide, and found 144.78 (O=16) as the atomic weight of didymium. By the aid of the Bunsen ice calorimeter he found that the specific heat of pure metallic didymium was 0.04563. This number multiplied by 144.78 gives 6.60, which agrees with the law of Dulong and Petit. This shows that the atomic weight of didymium must be expressed by a number very nearly of the order of that given above, and not by two-thirds of this amount. Therefore didymium forms the normal sesquioxide Di₂O₃, and not DiO.

Clève (*Bull. Soc. Chim.*, xxi., 246) heated the oxide in a stream of hydrogen to remove all superoxide, and from the sesquioxide thus obtained he synthesised the sulphate. As a mean of six determinations he obtained the value 147.01, maximum 147.23, minimum 146.65.

The presence of samarium was later discovered in the above material. This was removed (*Ibid.*, xxxix., 289), and ten new syntheses of the sulphate effected from the pure sesquioxide. The mean of these new results is 142.33, maximum 142.49, minimum 142.03.

Brauner (*Ber. der Chem. Ges.*, xv., 109; *Monatsh. Chem.*, iii., 14) effected the synthesis of the sulphate from the oxide, and, as the mean of three determinations, obtained the value 146.58.

Later (*Monatsh. Chem.*, iii., 499; *J. Chem. Soc.*, xliii., 278) he made five determinations with samples prepared in different ways, and found as a mean 145.42, with a difference between the highest and lowest value of 0.22. While these results agree fairly well with one another, they differ about a unit from his earlier result, which makes it probable that the oxide first employed contained some element of higher atomic weight. He tested this point as follows:—

Some of the didymium oxide used in the first series of experiments, which gave 146.58 as the atomic weight of the element, was fractionally precipitated with ammonia. The atomic weight of didymium, as determined with the fraction which was precipitated last, was found to be 145.40, which made it more probable that the substance first used contained an element with higher atomic weight. Further fractionation confirmed this point beyond question. Brauner concluded that the atomic weight of didymium was probably 145.2 to 145.4.

Bauer ("Inaugural Dissertation," Freiburg, 1884) synthesised the sulphate from the oxide, and obtained, as a mean of four determinations, the value 142.74.

Auer von Welsbach (*Monatsh. Chem.*, vi., 477) showed that didymium is not an element, but is composed of at least two, thereby invalidating all the preceding results, without, however, affecting the value of the methods which had been used. He effected the separation by

* From the *American Chemical Journal*, vol. xx., No. 5.

fractional crystallisation of the double nitrate of ammonium and didymium, from strong nitric acid, and repeating the process several thousand times. The one element forms green salts and green solutions, and the other rose-coloured solutions, and the salts are the colour of amethyst. To the former he gave the name *praseodymium*, and to the latter *neodymium*. Both the emission and absorption spectra of the two elements are different, but their sum is the same as the corresponding spectrum of didymium.

The elements resemble one another more closely than in any other known cases. Their conduct towards oxygen is, however, very different. Neodymium forms only the sesquioxide Nd_2O_3 , while praseodymium forms most readily the oxide Pr_2O_3 ; indeed this is formed whenever the oxalate, or sesquioxide, is heated in contact with air. It is easily reduced to the sesquioxide in a stream of hydrogen gas. Von Welsbach, without giving any details or even the method employed, assigned the atomic weight 143.6 to praseodymium, and 140.8 to neodymium.

Later, Crookes (*Nature*, xxxiv., 266) stated that, "although I have not split up didymium into the two earths, or groups of earths, which are described by Dr. Auer, other processes of fractionation gave me, so to speak, other cleavage planes, or lines of scission, through the compound molecule, didymium. . . . Neodymium and praseodymium must not be considered as actual chemical elements, but only the names given to two groups of molecules, into which the complex molecule didymium splits up, by one particular kind of fractionation."

The material used in carrying out the piece of work about to be described was furnished me, with unusual generosity, by Mr. Waldron Shapleigh, of the Welsbach Light Company, Gloucester, New Jersey, who has also made many valuable suggestions in connection with the separation of the elements. I wish to state here that this work was made possible only by the abundance of the comparatively pure praseodymium and neodymium compounds which Mr. Shapleigh placed at my disposal.

The Atomic Weight of Praseodymium.

About 1½ kilos. of the double nitrate of praseodymium and ammonium were furnished me by Mr. Shapleigh. It was analysed spectroscopically by means of the large Rowland spectroscope, with a concave grating of about 21 feet focal length, by Dr. W. J. Humphreys, and found to contain small amounts of neodymium, cerium, and lanthanum. No trace of any of the other rare elements, whose presence might reasonably be expected, could be detected.

The double nitrate, which had been thus far purified, was treated as follows:—The entire mass was dissolved in water containing a little nitric acid, and evaporated until crystals appeared. The solution was then cooled until a part of the double nitrate separated. This was filtered from the solution, and, since it contained more of the lanthanum, was discarded. The solution was then strongly acidified with nitric acid, and evaporated on the water-bath until, on cooling, about two-thirds of the total material crystallised out. This was the purest praseodymium, and was used for further crystallisation. The mother-liquor was filtered from these crystals and discarded, because it was richest in neodymium. The original substance was thus separated by fractional crystallisation into three parts, the first and last being discarded, and the second preserved as the purest material. The second portion was then treated exactly as the original substance, being separated into three portions by fractional crystallisation, and the middle portion again treated in the same manner. This process was repeated twenty-one times. Any impurities, such as calcium, iron, &c., were separated from the middle fraction of the last crystallisation, by precipitating the praseodymium with oxalic acid, in the presence of nitric acid. Only praseo-

dymium oxalate is thrown down under these conditions. The oxalic acid used was purified by repeated crystallisation from a mixture of alcohol and ether, to remove acid salts, and contained no detectable trace of either potassium or sodium. The water used in dissolving the praseodymium salt, also in washing the oxalate, and in all the processes of crystallisation, had been especially purified by distilling, first from acid permanganate, and then from alkaline, in the apparatus described by myself and Mackay (*Amer. Chem. Journ.*, xix., 91; *Zeitschr. Phys. Chem.*, xxii., 237). In all of this work the solutions were never allowed to come in contact with glass vessels while warm, but only with porcelain.

The precipitation of the oxalate was effected by pouring very slowly the hot dilute solution of the double nitrate of praseodymium and ammonium into a hot dilute solution of oxalic acid, with vigorous stirring. The oxalate thus precipitated separated in a form which could be easily washed free from impurities. A portion of this oxalate was subjected to spectrum analysis, and was found to contain a trace of neodymium, cerium, and lanthanum. The amount of the neodymium present was determined by comparing the intensity of the absorption bands of the neodymium in a solution of the praseodymium salt of known concentration, with the absorption bands of the neodymium salt, also of known concentration. A very concentrated solution of the double nitrate of praseodymium and ammonium was prepared, and the intensity of the absorption bands of neodymium noted. Then the solution of the double nitrate of neodymium and ammonium was diluted until the absorption bands were of the same intensity of those of the neodymium in the praseodymium salt. The amount of the neodymium in the solution of the neodymium salt was known, since this was prepared of standard concentration. The amount of the neodymium in the praseodymium salt could thus be calculated directly. By comparing the intensity of the absorption bands of neodymium in the two solutions, by bringing first the one and then the other into the field of the Steinheil spectroscopes, we could estimate very closely the amount of the neodymium present in the praseodymium salt. It was found to be 0.06 per cent, and could therefore be disregarded.

A further attempt was made to remove the trace of cerium and lanthanum, since the exact amounts present could not be determined accurately, because of the absence of sufficiently intense absorption bands. The oxalate of praseodymium, purified as just described, was decomposed by heat into the oxide, in a platinum vessel. The oxide was dissolved in nitric acid, the excess of acid evaporated, and the solution of the nitrate poured into a large volume of pure boiling water. The cerium present would then be precipitated as the basic nitrate, and a slight cloudiness in the solution was observed. This was repeated until the solution, in a large volume of water, was perfectly clear.

The solution of praseodymium nitrate was evaporated, and enough purified ammonium nitrate added to form the double salt. The double nitrate was then crystallised from a very strong nitric acid solution, far stronger than had been hitherto used, in order to remove the remaining trace of lanthanum. This was repeated several times.

The solution of the double nitrate of praseodymium and ammonium, thus purified, was poured into oxalic acid, after being acidulated with nitric acid, as before described, to remove any calcium, iron, &c., which might have gotten in during the separation of the traces of cerium and lanthanum. The oxalate was washed with the greatest care, converted into the oxide, and this again analysed spectroscopically by Mr. L. E. Jewell. No trace of cerium was detected, and only a trace of lanthanum.

This oxide, which was used in the following determinations, was blackish brown, and had the general properties of a superoxide. When dissolved in sulphuric acid there was a copious evolution of oxygen.

It was easily reduced to the sesquioxide when heated in a stream of hydrogen gas. When the superoxide was reduced to the sesquioxide it lost 2.5 per cent of oxygen, corresponding closely to the composition Pr_2O_7 , which is the composition of the superoxide described by Von Welsbach (*Monatsh. Chem.*, vi., 489). It is thus quite clear that the superoxide of praseodymium which I employed, agreed in all its essential properties with that isolated and used by Von Welsbach.

(To be continued.)

PROCEEDINGS OF SOCIETIES.

CHEMICAL SOCIETY.

Ordinary Meeting, June 2nd, 1898.

Professor DEWAR, F.R.S., President, in the Chair.

Messrs. A. L. H. Garside and K. J. P. Orton were formally admitted Fellows of the Society.

Certificates were read for the first time in favour of Messrs. Walter Birkett, 23, Cheviot Street, Lincoln; Alfred Hartridge, 14, Broad Street, Oxford; Charles William Tuthill Woods, St. Kilda, Tudor Road, Upper Norwood.

The Death of Lord Playfair.

The PRESIDENT said that the melancholy duty devolved on him to announce the death of the Right Hon. Lord Playfair, P.C., G.C.B., LL.D., F.R.S., the senior Past President and last surviving Founder of the Society, which took place on Sunday, May 29th. It was needless for him to dwell on Lord Playfair's scientific standing, or well recognised position in public life; he would only refer to the fact that he was the creator of a new type of public servant, and his services to education and as a member of Royal and Departmental Commissions on subjects connected with the public welfare were so unquestioned that it would be well for the country if there were more such men. Lord Playfair was ever ready to acknowledge his indebtedness to Liebig, Graham, and his other teachers, and proud to attribute his successful career to his early training in scientific method as a chemist. The Society would be represented at the funeral at St. Andrews by a former President, Professor Crum Brown, F.R.S.

In consequence of the death of Lord Playfair, the Banquet to the Past Presidents, arranged for June 9th, had been postponed until Friday, October 28th.

The following papers were read:—

83. "The Boiling-point and Density of Liquid Hydrogen." By JAMES DEWAR, LL.D., F.R.S.

The boiling-point of liquid hydrogen at atmospheric pressure has been determined by a platinum resistance thermometer. This was constructed of pure metal and had a resistance of 5.3 ohms at 0°C ., which fell to about 0.1 ohm when the thermometer was immersed in liquid hydrogen. On reduction of this resistance to normal air temperatures, the boiling-point is found to be -238.2° and -238.9° respectively by two methods, and to be -237° by a Dickson formula calculated for this thermometer (*cf. Phil. Mag.*, 1898, xlv., 525). The boiling-point of the liquid is, therefore, about -238°C . or 35° absolute, and is thus about 5° higher than that obtained by Olzewski by the adiabatic expansion of the compressed gas, and about 8° higher than that deduced by Wróblewski from van der Waals' equation. It may be inferred that the critical point of hydrogen is about 50° absolute, and that the critical pressure will probably not exceed 15 atmospheres. As molecular latent heats are proportional to absolute boiling-points, the latent heat of liquid hydrogen will be about two-fifths that of liquid oxygen. From analogy, it is probable that the practicable lowering of

temperature to be obtained by evaporating liquid hydrogen under pressures of a few m.m. cannot amount to more than 10 — 12°C ., and it may be said with certainty that no means are at present known for approaching nearer than 20 — 25° to the absolute zero of temperature. The platinum resistance thermometer used had a zero point of -263.2 platinum degrees, and when immersed in boiling liquid hydrogen indicated a temperature of -256.8° on the same scale, or 6.4 platinum degrees from the point at which the metal would become a perfect conductor. The effect of cooling platinum from the boiling-point of liquid oxygen to that of liquid hydrogen is to diminish its resistance to one-eleventh.

The approximate density of liquid hydrogen at its boiling-point was determined by measuring the volume of the gas obtained by evaporating to c.c., and is slightly less than 0.07, or about one-sixth that of liquid marsh gas, which has a density of 0.41, and is the lightest liquid at its boiling-point hitherto known. It is remarkable that, with so low a density, liquid hydrogen is so easily seen, has so well-defined a meniscus, and can be so readily collected and manipulated in vacuum vessels. As hydrogen occluded in palladium has a density of 0.62, it follows that it must be associated with the metal in some other state than that of liquefaction. The atomic volume of liquid hydrogen at the boiling-point is about 14.3, the atomic volumes of liquid oxygen and nitrogen being 13.7 and 16.6 respectively at their boiling-points. The density of the gas at the boiling-point of liquid hydrogen is 0.55, or about one-half that of air, and is eight times that of the gas at ordinary temperatures. The ratio of the density of hydrogen gas at the boiling-point to that of the liquid is approximately 1 : 100, as compared with a ratio of 1 : 255 in the case of oxygen.

The specific heat of hydrogen in the gaseous state and in hydrogenised palladium is 3.4, but may very probably be 6.4 in the liquid substance. Such a liquid would be unique in its properties; but as the volume of 1 gram. of liquid hydrogen is about 14—15 c.c., the specific heat per unit volume must be nearly 0.5, which is about that of liquid air. It is highly probable, therefore, that the remarkable properties of liquid hydrogen predicted by theory will prove to be susceptible of explanation when they are compared with those of liquid air, volume for volume, at corresponding temperatures as defined by van der Waals.

84. "The Action of Hydrogen Bromide in presence of Ether on Carbohydrates and certain Organic Acids." By HENRY J. HORSTMAN FENTON, M.A., and MILDRED GOSTLING, B.Sc.

It has been previously shown by one of the authors (*Trans.*, 1896, lxvii., 553) that ethylic dihydroxymaleate can be readily produced by the interaction of the acid with anhydrous ether in presence of hydrogen bromide. This behaviour of ether as a "base" being unusual, at any rate, towards organic acids, it was considered desirable to make experiments with other acids in a similar manner; the results indicate that the reaction is a general one, and that the yields are satisfactory.

The study of the behaviour of ether and hydrogen bromide is now being extended to substances other than acids, and an account is given of the results obtained with carbohydrates and allied polyhydric alcohols. Certain of these substances develop an intense purple colouration, and it is found that the presence of ether, although advantageous, is not essential for its production. The coloured substance seems to resemble the compound described by Stanhouse and others as "metafurfural." So far as the investigation has proceeded, ketohexoses and carbohydrates such as inulin, which yield them on hydrolysis, give an intense purple colouration in the course of an hour or two; aldohexoses give a purple colouration of comparatively moderate intensity in the course of a day or two; and other carbohydrates, such as arabinose, rhamnose, starch, and cellulose, and alcohols such as mannitol, dulcitol, and glycerol, give a brown, red, or

yellow colouration free from any shade of purple. The only exception to this classification yet met with is xylose, which, although in the purest state obtainable, behaves similarly to the ketohexoses.

DISCUSSION.

Mr. CHAPMAN asked whether the authors had made any experiments with hydrogen chloride. Bouillon-Lagrange and Vogel (*Annales de Chimie*, 1809, lxxi., 91) had stated that both cane-sugar and milk-sugar absorb hydrogen chloride, forming coloured products, which evolve the gas with effervescence on treatment with sulphuric acid.

Mr. FENTON, in reply, said that hydrogen chloride gives only a slight red colour, even after long standing, and that hydrogen iodide acts as a reducing agent liberating iodine.

85. "Production of some Chloropyridinecarboxylic Acids." By J. N. COLLIE, Ph.D., F.R.S., and W. LEAN.

The authors have prepared from ethylic chlorolutidine-carboxylate (*Trans.*, 1897, lxxi., 303) several chloropyridinecarboxylic acids, and, by reducing one of them with tin and hydrochloric acid, carbocinchomeric acid, which is also one of the products of the action of oxidising agents on quinine, and several other alkaloids.

Ethylic chlorolutidinemonocarboxylate was prepared by the action of phosphorus pentachloride on ethylic oxy-lutidine carboxylate. It is a sweet-smelling liquid, boiling at 288–290°. Chlorolutidinecarboxylic acid can be obtained by hydrolysis of the ethylic salt; it crystallises in small needles, melting at 148°. By the action of potassium permanganate on the ethylic salt, two acids were obtained, one having the formula—



and melting at 169°, the second being an α' -chloropyridine- $\alpha\beta$ -tricarboxylic acid, $C_7HN(CO_2H)_3Cl$, melting at 212°. The latter acid yielded carbocinchomeric or pyridine, $\alpha\beta$ -tricarboxylic acid, $C_7H_2N(CO_2H)_3$, when reduced with tin and hydrochloric acid.

PHYSICAL SOCIETY.

Ordinary Meeting, June 10th, 1898.

Mr. SHELFORD BIDWELL, President, in the Chair.

Dr. S. P. THOMPSON described and exhibited a model illustrating Max Meyer's theory of Audition. Max Meyer abandons the audition theory of Helmholtz, and contends that analysis takes place in the ear otherwise than by resonance of the Corti organ. Imagine a jointed system, like a hand, to be oscillated from one end, i.e., from the finger-tips. A small motion affects only the top joints, but a large motion affects the whole structure. Such a structure is the membrane of the inner-ear. It widens towards one end, and is effectively damped by the contained liquid. Wave-motions of different amplitudes run along it to different distances before they are extinguished; these distances are recorded by nerves, and are thereby communicated to the Corti organ. In the model the compound-wave to be analysed is cut out on the edge of a disc of zinc, so that, as the disc revolves, the motions are communicated to a frame-work. If the frame is thus moved through more than a certain distance, a displacement occurs which sets a second frame in motion, and so on to a third and fourth. The depth to which the motion penetrates is indicated by a series of glow-lamps connected electrically to the frames.

Prof. Ayrton said it had for some time past occurred to him, when considering the way in which an expert telegraph clerk reads syphon-recorder signals on a long cable, that it might be possible to analyse waves without the supposition of a resonating apparatus. The clerk interprets not so much the motions to one side or other of the zero-line as the rate of change of velocity, i.e., the

acceleration of the syphon. This had been recognised in the design of those relays for long cables, where the lever makes contact when the received current exceeds a certain value, and breaks contact when the current falls below a certain minimum. Messrs. Siemens had adopted a relay in which the lever was carried on the suspended coil of a d'Arsonval galvanometer by a pivot with a small amount of friction. If contact was made, the coil could, nevertheless, continue its motion in a given direction. If that direction altered, contact was immediately broken, and the lever passed over to the opposite stop, thereby reversing the local circuit. It was possible that, in the process of hearing, something akin to this took place, the ear behaving as a mechanism responsive, not by resonance to the complete waves, but by its sensitiveness to changes of direction of the received impulses.

Dr. S. P. THOMPSON thought that a mechanism similar to the relay described by Prof. Ayrton was contained in the telautograph of Elisha Gray; it was a "Prony" mechanism. In the acoustical problem the ear was probably sensitive to abrupt changes of shape in the waves as well as to reversals. In the case of mistuned octaves, something is heard that suggests "revolving" in the ear, indicating a cyclic change. In this regard it was necessary to take into account the phase-relations as well as the relative intensities of the component tones.

Mr. E. H. BARTON then read a paper on the "Attenuation of Electric Waves along a Line of Negligible Leakage."

It forms a sequel to a paper communicated to the Physical Society and printed in their *Proceedings* of December, 1897, and January, 1898. Shortly after the publication of the earlier results, Mr. Oliver Heaviside drew attention to Lord Rayleigh's high-frequency formula for the "Effective Resistance" of wires to alternating currents, and suggested that the formula might be approximately applicable to the case; but he thought the experimental value of the attenuation would be considerably higher than the one derived from calculations. Mr. Barton here repeats the work, with special precautions as to the mode of insulating the parallel copper wires through which the wave-train proceeds. The value of the attenuation-constant deduced from these experiments is 0.00003. By applying Lord Rayleigh's formula for the effective resistance of the circuit, and using this value in Mr. Heaviside's expression for the attenuation, the calculated constant is 0.0000062. To account for the discrepancy, the author points out that the effective resistance formula was originally developed for a wire placed at a considerable distance from other parts of the circuit, and for currents following the harmonic law. Whereas, in the experiments the conditions are—(1) wires 1.5 m.m. diameter, only 8 c.m. apart, and (2) the waves are propagated in the form of a damped train, with the large end leading; they are extinguished after ten or a dozen vibrations.

Mr. OLIVER HEAVISIDE (communicated) pointed out that, as there was human interest in error, it might be worth mentioning that at first it was supposed the previous experiments of Dr. Barton made the index of the attenuation-factor to be six times that of the long-wave theory for simple periodic waves. And it was hard to account for so large a discrepancy. The discovery of an error in the figures reduced the result from six to two. The small depth of the surface-layer of effective conduction, and the distance apart of the wires, seemed now to make it improbable that E. Barton's first reason, (1), was adequate to account for the doubling of resistances. The second, (2), was of course a substantial reason for increased resistance. A third one, Mr. Heaviside suggested, was the external resistance at the boundary of the waves. A combination of the second and third reasons, with a little of the first, might account for most of the extra attenuation observed, and, if more was wanted, one could "try the K. R. law."

Mr. APPEYARD said it was rather to be regretted that, in all the experiments, the distance between the wires had been the same, i.e., 8 c.m. By taking a few different values (1) might have been checked. Lord Rayleigh's formula for the effective resistance involved the square-root of the magnetic permeability of the wires. The author had, throughout, used copper, a paramagnetic metal, and had assumed $\mu = 1$. It would be of advantage to try other metals.

Mr. BARTON, in reply, said he would make further experiments with the two conductors at different distances apart, and he would also try iron wires. With iron, the thickness of the surface-layer of the effective conductor was about one-thirteenth that of copper. Iron should therefore give a greater value of the attenuation than copper.

Mr. A. GRIFFITHS then read a paper on "*Diffusive Convection*,"—a phenomenon analogous to caloric convection.

The differences of density that produce convection-currents are not due to changes of temperature, but to variations in the quantity of dissolved substance per unit volume. The author has devised an apparatus consisting of a vessel divided horizontally by a diaphragm, through which pass two vertical tubes of unequal lengths. A solution of copper sulphate, maintained at constant strength, is placed in the lower compartment. The upper compartment is filled with water. Diffusion takes place up the tubes. One tube is 4 c.m. long, the other is 4'05 c.m. The tops of the tubes are exactly at the same level. Up the longer tube, and down the shorter, diffusive convection occurs at the rate of 5 c.m. per year. This flow *increases* the quantity of copper sulphate transmitted by the long tube by about 2 per cent, and *diminishes* that transmitted by the shorter tube by about the same amount. Consequently, the resultant increase due to the motion is only a fraction of 1 per cent. To detect the flow the author employs a second piece of apparatus, in which the upper ends of the tubes are separated by a capillary, containing coloured liquid. By this means the motion is considerably magnified.

Dr. S. P. THOMPSON asked whether, in a case where a large tube was used in determining the velocity, the viscosity of the liquid would not play a very much less part than with narrow tubes.

Mr. GRIFFITHS explained that viscosity was not important until very small tubes were considered, e.g., those of the order 0'001 m.m. diameter.

The PRESIDENT proposed votes of thanks to the authors, and to Dr. Max Meyer, for lending the Society his model. The meeting then adjourned until June 24th.

CORRESPONDENCE.

GAS AND GASES.

To the Editor of the Chemical News.

SIR,—Attentive examination of the statement made to the Royal Society, by Prof. Ramsay, at the meeting of June 9th, shows that, instead of establishing a new element, he has disestablished one already accepted. It is obvious that the residual gas obtained from the atmosphere, by repeating the classical experiment of Cavendish, is a mixture of argon and krypton in unknown proportions. It is equally obvious that the residual gas obtained from the evaporation of the last few cubic centimetres of a quantity of liquid air is a mixture of the same gases, again in unknown proportions. The first may be presumed to contain more argon than krypton, and the second more krypton than argon; but that is all that can be said with any degree of certainty.

Professor Ramsay talks of disentangling the spectra of

these two gases, by the simple process of assigning to krypton the lines that do not belong to argon. But as he has never isolated argon he does not know what lines belong to it and what lines to the admixture of krypton. He is simply comparing two mixtures in different proportions, but in neither case in known proportions, one of which at least must be of variable composition. In such circumstances it is evident that argon and krypton are mere names, connoting no scientific entity whatever. For anything Professor Ramsay knows, what he now calls krypton may be a mixture, just as he has proved that what he called argon is a mixture. Indeed it is practically certain that a third gas is present in the Cavendish residue, because some gas not yet named has been escaping for centuries from the Bath springs along with nitrogen and helium.

Now, Sir, I submit that to claim the discovery of new elements on such grounds as these is not science. If a man is to be hailed as the discoverer of a new element merely because, from a mixture which he has not analysed, he obtained a spectroscopic line or lines not yet assigned to a known element, then Liveing and Dewar discovered krypton four years ago, and Dewar discovered last November an element to which he has not given a name. Cavendish proved, more than a century ago, that the air contains something which is neither oxygen, nitrogen, nor carbonic acid. Except that the spectroscope proves this something to be more complex than Cavendish would probably have supposed it, or that Professor Ramsay supposed it when he announced it to be argon, I am totally at a loss to see what addition of accurate scientific knowledge has been made to what Cavendish proved.—I am, &c.,

SUUM CUIQUE.

NOTICES OF BOOKS.

Optical Activity of Organic Substances, and its Applications. ("Das Optische Drehungsvermögen Organischer Substanzen und dessen Praktische Anwendungen.") By Dr. H. LANDOLT, Professor of Chemistry in the University of Berlin. Second Edition, completely Revised. Pp. 655. Brunswick. 1898.

SINCE the first edition of this work, in 1879, the interest of chemists in the subject of "optical activity" of organic substances has received a great stimulus from the acceptance of the theory of the asymmetric carbon atom—a theory which at that time was receiving the first confirmations of practical experiment.

From the time that van't Hoff and Le Bel pointed out the now undoubted relationship between optical activity and atomic constitution in organic compounds, the records of research in this branch of chemistry have accumulated to bewildering proportions.

To reduce to order a mass of literature covering a period of nearly twenty years' work on this subject was by no means an easy task. That the author has been completely successful must be the opinion of all chemists who read the book, and who are interested in the subject upon which it bears:

To the practical worker the book will be invaluable, methods of investigation being admirably described, and details of manipulation completely given.

For convenience the book is divided into six parts. The first part is devoted to the general considerations of optical activity. The history of the subject is briefly touched upon; then come "Classification of Active Bodies," "Nature of Optical Activity," "Relationship between Optical Activity and Chemical Constitution of Organic Substances," and "Optical Modifications." This last chapter of Part I. gives a very complete account of the more important optical isomers, with tabulated details

of their relative densities, physiological action, solubility, &c.

The second part gives an account of the Laws of Circular Polarisation, and the third part treats of the numerical values for the optical activity of substances under varying conditions (*e.g.*, when crystalline, and in concentrated and dilute solutions), and of the influence of solvents, &c.

The fourth part gives details of apparatus and methods for the determination of Specific Rotation. The fifth part treats of the "Practical Applications of Optical Activity," the subject of saccharimetry receiving special attention.

Part the sixth, and last, gives a *resumé* of the numerical values so far found for the rotatory power of optically active bodies.

The theories bearing on the subject of the work are clearly and concisely stated. The tables throughout will save endless reference, and must have cost a vast amount of labour and care. We strongly recommend the book to all serious students of chemistry.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Comptes Rendus Hebdomadaires des Séances, de l'Académie des Sciences. Vol. cxxvi., No. 17, April 25, 1898.

Industrial Treatment of the Emerald by the Electric Furnace.—B. Lebeau.—If this mineral is heated in a carbon tube in the electric furnace for eight or ten minutes with a current of 950 ampères and 45 volts, the greater part of the silica distils off, and there remains a melted mass having a metallic lustre; this consists of a mixture of carbide of aluminium, carbide of glucinum, silicide of iron, and silicide of carbon. It is attacked by dilute acids, giving a solution containing glucina and alumina. By using hydrofluoric acid a fairly pure solution of fluoride of glucinum can be obtained at once, fluoride of aluminium being insoluble. If half its weight of carbon is added to the emerald a complete reduction is soon arrived at; the silica is completely eliminated either by volatilisation or by the formation of silicide of carbon, a body almost as unassailable as diamond. Some of these experiments were carried out on 100 kilos. of emerald.

On the Quinoximes.—A. Valeur.—The quinones form two kinds of oximes, the mono-oximes, and the di-oximes. The first may be obtained by the action of chlorhydrate of hydroxylamine on the quinones in alcoholic solution, or by the action of nitrous acid on the phenols; the quinoximes are, in fact, identical with the nitroso-phenols. In this paper the author gives the results of his examination of the monoximes of ordinary quinone, of thymoquinone, and of the naphthoquinones. He has measured the heat of combustion of a certain number of these compounds, and finds a constant difference between the heat of combustion of these oximes and the corresponding quinones of about 60 cal.

On the Products from the Splitting-up of Ouabaine by Hydrolysis.—M. Arnaud.—Boiling dilute hydrochloric and sulphuric acids hydrolyse ouabaine completely, though slowly. The splitting-up is first shown by the cloudy appearance of the liquid, which becomes milky, and finally gives a rapidly increasing deposit of a deep yellow resinous matter. The hydrolysis is generally done on 20 grms. of crystallised hydrated ouabaine, $C_{30}H_{40}O_{12} \cdot 9H_2O$, dissolved in twelve times its weight of water acidulated with two per cent of H_2SO_4 , heated in a sealed tube at 100° for forty or fifty hours. The clear liquid, separated from the melted residue, is neutralised with baryta. The esti-

mation of the reducing sugar is made by means of Fehling's solution, and compared with a typical solution of pure rhamnose. It is found that 21.70 to 21.80 per cent of anhydrous rhamnose, $C_6H_{12}O_5$, corresponds to the hydrated ouabaine; the identity of these two bodies has been established. The resin has not been obtained in crystals; it is easily soluble in concentrated alcohol, methyl alcohol, ether, &c. To obtain it in the anhydrous state it must be heated to above its melting point, 130° — 135° , away from contact with air, as it is very readily oxidisable when warmed. The splitting-up of ouabaine is expressed thus:—



Chloridising Action of Ferric Chloride in the Aromatic Series.—V. Thomas.—The reduction of ferric chloride by benzene commences at the ordinary temperature; though the quantity of hydrochloric acid set free in the cold is small, the reduction is more rapid when the temperature is raised, and at 80° it is very energetic. When this action is ended the residue is exhausted with boiling benzene and distilled; between 130° and 134° a colourless liquid comes over—this is monochlorobenzene. This is not the extent of the action; by starting with more or less chloridised benzenes (mono-, di-, tri-, &c.) the whole series up to the hexachloride can be obtained. A similar reaction takes place with the toluene.

The Phosphoric Di-Ethers.—J. Cavalier.—The author has shown that when phosphoric acid, PO_4H_3 , passes to the state of mono-ether, PO_4RH_2 , it is the acidity of the weakest which disappears. The results here show that the same is the case when a new radical, R, is introduced, when passing from PO_4RH_2 to the di-alcoholic acid, PO_4R_2H . The acid function which persists is comparable with the most energetic of the acid functions of the mono-alcoholic acid and the phosphoric acid.

The Acid Phosphoglycerates.—MM. Adrian and Trillat.—The acid phosphoglycerates can be prepared by decomposing the corresponding neutral salts by the theoretical amount of sulphuric acid, in the presence of heli-antine as an indicator, or by the double decomposition between the acid salt of barium and a soluble sulphate. The acid phosphoglycerates are distinguished from the neutral salts by their great solubility in water. They are difficultly precipitable from their solutions by alcohol, which even at 50° dissolves a considerable proportion. They do not crystallise, and when dried *in vacuo* they appear in the form of a white vitreous mass (blue in the case of the copper salt), unchangeable at 100° . On the other hand their aqueous solutions are decomposed on boiling, forming free phosphoric acid and glycerin.

MISCELLANEOUS.

The Kekulé Memorial.—We desire to recall the attention of our readers to the Kekulé Memorial Fund, as to which an announcement appeared in our columns on February 25th last (CHEM. NEWS, vol. lxxvii., p. 88), and to urge any who intend to subscribe to send in their contributions without delay to Dr. Hugo Müller, 13, Park Square East, N.W., as we understand that the Committee propose shortly to close the account.

Paris Exhibition of 1900.—The Royal Commission are now prepared to circulate information respecting the Exhibition. The classification and rules for Exhibitors, together with forms of application for space, can be obtained by applying to the Secretary of the Royal Commission, Paris Exhibition 1900, St. Stephen's House, Westminster, S.W.

Royal Institution.—A General Monthly Meeting of the Members of the Royal Institution was held on Monday afternoon (the 6th inst.), Sir James Crichton-Browne,

M.D., F.R.S., Treasurer and Vice-President, presiding. The following was elected a Member:—Mr. A. W. Horsburgh. The special thanks of the Members were returned for the following donations to the fund for the promotion of Experimental Research at Low Temperatures:—Mrs. G. J. Romanes, £5; Sir Frederick Bramwell, £100; Professor Dewar, £100; Dr. Ludwig Mond, £200; Charles Hawksley, Esq., £100; Sir David Salomons, Bart., £21; Dr. Rudolph Messel, £100.

Investigation of Crystalline Liquids.—R. Schenck. —The author proves that liquid crystals correspond, not only in their optical, but also in their whole behaviour, to solid crystals. In liquid crystals there is a changing point, which in all cases expresses a melting point. He examines the behaviour of the liquids *p*-azoxyanisole, *p*-azoxyphenetole, and cholesteryl benzoate, by estimating the surface energy at various temperatures, from measurements of the density of the isotropic and anisotropic phases of the liquid.—*Zeit. für Physikalische Chemie*, xxv., No. 2.

An International Competition for the invention of a paste containing no white phosphorus, to be used in the manufacture of matches, is being organised by the Belgian Government. A prize of £2000 is offered to the successful competitor. The following are some of the conditions governing the competition:—The paste must offer such resistance to shocks and friction that dangerous explosions need not be feared during manufacture, and it must not contain any material which may be dangerous to the health of the operatives. The matches must be capable of igniting when rubbed on various substances, even on cloth, and must be able to withstand various alterations of moisture and dryness, heat and cold, within certain limits, without deterioration; they must not be spontaneously inflammable or decomposable. Inventors will be admitted to the competition until January 1st, 1899. The successful inventor must be able to furnish proof that the manufacture of his matches is commercially possible, and must engage to manufacture at least one million matches on Belgian territory in the presence of the Commissioners or their delegates. Further particulars of the competition can be obtained from M. Woeste, Ministère de l'Industrie et du Travail, 2, Rue Laterale, Brussels.

On Galacyl.—C. André.—It is well known that local anaesthesia can be produced by means of galacal in subcutaneous injections, and the author determined to try the effect of galacyl,—that is, the calcium derivative of galacyl-sulphuric acid, $(C_7H_7O_2SO_3)Ca$, in collaboration with Dr. O'Follioinel. He (the author) placed 100 grms. of pure galacal in a matras, and gently heated it; when liquid he added very gradually 100 grms. of pure concentrated sulphuric acid, and left it at the ordinary temperature for 48 hours. It was then diluted with six or seven times its weight of distilled water, and saturated with carbonate of lime in small pieces, at 80° on the water-bath; it was then filtered and evaporated to dryness, and taken up in four or five times its weight of alcohol at 90°; this separates out a small quantity of insoluble substances. After re-evaporation and pulverisation a greyish mauve powder is left, very soluble in water and alcohol, but not in oil; this is galacyl. For anaesthesia 1/10th to 1/20th solutions of galacyl in water were used; they gave practically the same results. In extracting teeth, out of thirty-two cases, in twenty-two the anaesthesia was sufficient, in seven it was incomplete, and twice there was no effect—one case was doubtful. Out of eleven small surgical operations, in five the anaesthesia was complete, five times it was sufficient, and once it failed; that is to say, 90 per cent of success.—*Journ. de Pharm. et de Chemie*, Series 6, vol. vii., No. 7.

Synthesis of Terebic Acid.—E. E. Blaise.—The author has obtained 10 to 15 grms. per cent of terebic acid by the condensation of bromosuccinate of ethyl in an equal-molecular mixture with acetone, to which an equal weight of bromised ether was added.—*Bull. Soc. Chim. de Paris*.

NOTES AND QUERIES.

. Our Notes and Queries column was opened for the purpose of giving and obtaining information likely to be of use to our readers generally. We cannot undertake to let this column be the means of transmitting merely private information, or such trade notices as should legitimately come in the advertisement columns.

Ozonator.—I notice in your last issue that a correspondent wishes to know where he can find particulars of my ozonisers. He may write or call at the Electric Ozone Syndicate, Ltd., 5, New Union Street, Moorgate St. Station, E.C.—E. ANDREWS.

MEETINGS FOR THE WEEK.

WEDNESDAY, 22nd.—Institution of Mining and Metallurgy, 2. "The Treatment of Broken Hill Complex Sulphide Ores by Wet Extraction Processes and the Electrolytic Deposition of Zinc," by Edgar A. Ashcroft, M.Inst.M.M. "Notes on the Payable Conglomerate Beds encased in Sandstone, and the Mining Methods in use for their extraction on the Witwatersrand," by A. F. Croose, M.Inst.M.M. "Density of Gold and Silver Alloys," by George Attwood, M.Inst.M.M.

FRIDAY, 24th.—Physical, 5. Exhibition of an Apparatus illustrating the Action of Two Coupled Electric Motors by Prof. Carus-Wilson. Exhibition of Weedon's Expansion of Solids Apparatus by Mr. J. Onick. "Theory of the Hall Effect in a Binary Electrolyte," by F. G. Donnan, M.A., Ph.D.

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CONTENTS.

ARTICLES:—	PAGE
On a New Constituent of Atmospheric Air, by William Ramsay, F.R.S., and Morris W. Travers.....	287
Note on the Estimation of Rosin and Rosin Oil in Linseed Oil, by L. de Koningh, F.I.C., F.C.S.....	287
Spectral Research on Atmospheric Air, by MM. Moissan and Deandres.....	288
On the Preparation and Properties of Anhydrous Fluoride of Glucinum and of Oxyfluoride of Glucinum, by P. Lebeau....	288
On a Borocarbide of Glucinum, by P. Lebeau.....	289
On the Absorption of Nitric Oxide by Ferrous Salts, by V. Thomas.....	290
A Determination of the Atomic Weight of Praseodymium and of Neodymium, by Harry C. Jones.....	292
The Action of Carbon Dioxide on Soluble Borates, by Louis Cleveland Jones.....	294
NOTICES OF BOOKS.—Laboratory Experiments on the Glass Reactions, and Identification of Organic Substances—French Self-taught, with Phonetic Pronunciation—&c.....	296
CORRESPONDENCE.—The New Gases.....	297
CHEMICAL NOTICES FROM FOREIGN SOURCES.....	297

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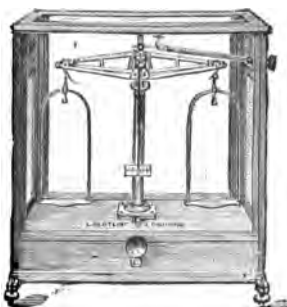
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THE CHEMICAL NEWS.

VOL. LXXVII., No. 2013.

ON A

NEW CONSTITUENT OF ATMOSPHERIC AIR.*

By WILLIAM RAMSAY, F.R.S. and MORRIS W. TRAVERS.

THIS preliminary note is intended to give a very brief account of experiments which have been carried out during the past year of attempts to ascertain whether, in addition to nitrogen, oxygen, and argon, there are any gases in air which have escaped observation owing to their being present in very minute quantity. In collaboration with Miss Emily Aston we have found that the nitride of magnesium, resulting from the absorption of nitrogen from atmospheric air, on treatment with water yields only a trace of gas; that gas is hydrogen, and arises from a small quantity of metallic magnesium unconverted into nitride. That the ammonia produced on treatment with water is pure has already been proved by the fact that Lord Rayleigh found that the nitrogen obtained from it had the normal density. The magnesia, resulting from the nitride, yields only a trace of soluble matter to water, and that consists wholly of hydroxide and carbonate. So far, then, the results have been negative.

Recently, however, owing to the kindness of Dr. W. Hampson, we have been furnished with about 750 c.c. of liquid air, and, on allowing all but 10 c.c. to evaporate away slowly, and collecting the gas from that small residue in a gas-holder, we obtained, after removal of oxygen with metallic copper, and nitrogen with a mixture of pure lime and magnesium dust, followed by exposure to electric sparks in presence of oxygen and caustic soda, 26·2 c.c. of a gas, showing the argon spectrum feebly, and, in addition, a spectrum which has, we believe, not been seen before.

We have not yet succeeded in disentangling the new spectrum completely from the argon spectrum, but it is characterised by two very brilliant lines, one almost identical in position with D_3 , and almost rivaling it in brilliancy. Measurements made by Mr. E. C. C. Baly, with a grating of 14,438 lines to the inch, gave the following numbers, all four lines being in the field at once:—

D_1	..	..	..	5895·0
D_2	..	..	..	5889·0
D_3	..	..	..	5875·9
D_4	..	..	..	5867·7

There is also a green line, comparable with the green helium line in intensity, of wave-length 5568·8, and a somewhat weaker green, the wave-length of which is 5560·6.

In order to determine as far as possible which lines belong to the argon spectrum, and which to the new gas, both spectra were examined at the same time with the grating, the first order being employed. The lines which were absent, or very feeble, in argon, have been ascribed to the new gas. Owing to their feeble intensity, the measurements of the wave-lengths which follow must not be credited with the same degree of accuracy as the three already given, but the first three digits may be taken as substantially correct:—

Violet ..	..	4317	Blue ..	..	4834
" ..	..	4387	" ..	..	4909
" ..	..	4461	Green ..	..	5560·6
" ..	..	4671	" ..	..	5568·8
Blue ..	..	4736	Yellow ..	..	5829
" ..	..	4807	" ..	..	5867·7
" ..	..	4830	Orange ..	..	6011

* A Paper read before the Royal Society, June 9th, 1898.

Mr. Baly has kindly undertaken to make a study of the spectrum, which will be published when complete. The figures already given, however, suffice to characterise the gas as a new one.

The approximate density of the gas was determined by weighing it in a bulb of 32·321 c.c. capacity, under a pressure of 521·85 m.m., and at a temperature of 15·95°. The weight of this quantity was 0·04213 grm. This implies a density of 22·47, that of oxygen being taken as 16. A second determination, after sparking for four hours with oxygen in presence of soda, was made in the same bulb; the pressure was 523·7 m.m., and the temperature was 16·45°. The weight was 0·04228 grm., which implies the density 22·51.

The wave-length of sound was determined in the gas by the method described in the "Argon" paper. The data are:—

	i.	ii.	iii.
Wave-length in air ..	34·17	34·30	34·57
" " gas ..	29·87	30·13	

Calculating by the formula,—

$$\lambda^{\text{air}} \times \text{density air} : \lambda^{\text{gas}} \times \text{density gas} :: \gamma_{\text{air}} : \gamma_{\text{gas}},$$

$$(34·33)^{\text{a}} \times 14·479 : (30)^{\text{a}} \times 22·47 :: 1·408 : 1·666,$$

it is seen that, like argon and helium, the new gas is monatomic, and therefore an element.

From what has preceded, it may be concluded that the atmosphere contains a hitherto undiscovered gas with a characteristic spectrum, heavier than argon, and less volatile than nitrogen, oxygen, and argon; the ratio of its specific heats would lead to the inference that it is monatomic, and therefore an element. If this conclusion turns out to be well substantiated, we propose to call it "krypton" or "hidden." Its symbol would then be Kr.

It is, of course, impossible to state positively what position in the periodic table this new constituent of our atmosphere will occupy. The number 22·51 must be taken as a minimum density. If we may hazard a conjecture, it is that krypton will turn out to have the density 40, with a corresponding atomic weight 80, and will be found to belong to the helium series, as is, indeed, rendered probable by its withstanding the action of red-hot magnesium and calcium on the one hand, and on the other of oxygen in presence of caustic soda, under the influence of electric sparks. We shall procure a larger supply of the gas, and endeavour to separate it more completely from argon by fractional distillation.

It may be remarked in passing that Messrs Kayser and Friedländer, who supposed that they had observed D_3 in the argon of the atmosphere, have probably been misled by the close proximity of the brilliant yellow line of krypton to the helium line.

On the assumption of the truth of Dr. Johnstone Stoney's hypothesis that gases of a higher density than ammonia will be found in our atmosphere, it is by no means improbable that a gas lighter than nitrogen will also be found in air. We have already spent several months in preparation for a search for it, and will be able to state ere long whether the supposition is well founded.

NOTE ON THE ESTIMATION OF ROSIN AND ROSIN OIL IN LINSEED OIL.

By L. DE KONINGH, F.I.C., F.C.S.

THE excellent method devised by Parker C. McIlhenny (*J. Amer. Chem. Soc.*, xvi., 275—278) for the detection of the adulteration of boiled linseed oil has not been so universally adopted as it deserves; this is, no doubt, caused by the fact that it does not distinguish between rosin and rosin oil.

By combining this process—for the details and figures of which the original paper should be consulted—with

the method for extracting unsaponifiable oils from soapy solutions by means of light petroleum, a very good assay for the purity of linseed oil is at disposal.

Samples of rosin oil used in this country for the adulteration of linseed oil were found by the writer to contain not more than 5 per cent of saponifiable matter, but Fahrion (*Zeit. Angew. Chem.*, 1898, 270) has come across a sample containing nearly 20 per cent of the same. Linseed oil adulterated with such a sample will, therefore, appear to contain added rosin as well.

The writer's process is, then, very shortly as follows:—The bromine-acidity figure of the sample is taken. If the amount of unsaponifiable matter comes to about 5 per cent, and betrays the presence of rosin oil by its odour, its bromine-acidity figure is also taken. It does not do to liberate the acids from the extracted soap-ley, and then to take their bromine-acidity number; then, even when free from rosin, they will often give an acidity figure high enough to account for 5 per cent of this substance. The writer believes that this may be accounted for by the water of hydration. McIlhenny warns against the presence of free moisture; if this causes acidity; might not the combined water act still more energetically?

If the original sample gives only a slight acidity, this may be caused by the presence of free fatty acids, but a strong bromine-acidity may be put down to joint rosin-oil and rosin, part of which, however, may be derived from the rosin oil supposing any of the latter is found in the unsaponifiable matter. Actually added rosin is generally added as a metallic resinate, so if there is no ash the presence of true rosin becomes very doubtful.

The writer has had no opportunity of taking the bromine-acidity figure of such samples as mentioned by Fahrion, after alkali treatment; investigation in this respect is highly desirable.

4, St. Martin's Road, Stockwell, S.W.,
June 8, 1898.

SPECTRAL RESEARCH ON ATMOSPHERIC AIR.

By MM. MOISSAN and DESLANDRES.

At the meeting of the French Academy on the 15th of June last MM. Moissan and Deslandres asked for a sealed packet, deposited by them on May 11th, 1896, to be opened. This packet was numbered 5213, and was duly opened by the President. It contained the following note:—

During the course of researches undertaken on the gases given off by the mineral cerite, when heated *in vacuo*, we have carefully studied, by spectroscopic means, the residue left by these gases, after explosion with oxygen in the eudiometer and absorption of the excess of oxygen by means of pyrogallous acid.

The residue, (1 or 2 c.c.) was introduced into a vacuum tube connected with a mercury pump, fitted with drying tubes filled with caustic potash and anhydrous phosphoric acid.

Now the spectrum of the residue, examined principally in the blue and the violet, showed the characteristic bands of nitrogen fairly strongly, the helium lines, some of the argon lines, and, further, the following lines, which have not yet been ascribed to any known gas:—

Intensities counted from 1 to 10, 10 being the strongest.	Wave-lengths.
7	415'17
3	414'37
4	411'00
4	410'80
2	410'05

In this experiment the gas from the cerite and the oxygen were present in about equal quantities; but the residue obtained with oxygen only, also gave the new

lines more or less strongly, even when the oxygen had been prepared from chlorate of potash, permanganate of potash, and native binoxide of manganese.

However, the characteristic lines of nitrogen were always present; and as nitrogen was, to a certain extent, supplied by the use of the mercury oven, it was thought that atmospheric air might be playing a rôle, and so ordinary air was introduced into the same sparking-tube, and the spectra examined at diminishing pressures.

Now these new lines were still apparent, but only at a certain pressure, less than 1 m.m., and below the pressure of maximum brightness of the nitrogen tubes. At this same pressure the characteristic lines of argon would also be seen, though faintly, the line λ 415'87 being the most prominent.

The new lines appeared stronger when the air was replaced by nitrogen obtained from nitrite of ammonium, or separated from ordinary air by the usual pyrogallous acid method, the protochloride of chromium method, or by copper at a red heat.

On the other hand, the lines disappeared at the same time as those of nitrogen, when the nitrogen was absorbed by lithium or by magnesium. But still, these new lines might be strictly attributed to impurities, to vapours emitted, or to the matter actually forming the rarefied tube,—such as glass, metallic electrodes, mercury, grease, potash, phosphoric acid; but their relative intensity does not increase when the pressure gradually diminishes, as is the case with the mercury lines. Further, they have been obtained in spectrum tubes of crystal and of German glass, with and without metallic electrodes; they have been observed without the characteristic cyanogen bands, which form a very delicate test in the simultaneous presence of carbon and of nitrogen; they have again been persistent after the introduction of oxygen which caused the disappearance of all the mercury lines. Finally, they were missing in the gases which had been absorbed by potash and heated in the vacuum tube with either phosphoric acid or red or white phosphorus.

It may be concluded that these are either nitrogen lines due to low pressure and not yet noted, or that they are due to a new gas existing in the atmosphere, closely resembling nitrogen in its chemical properties. The simultaneous appearance of the argon lines render the latter alternative most probable.—*Comptes Rendus*, cxxvi., No. 24, June 15, 1898.

ON THE PREPARATION AND PROPERTIES OF ANHYDROUS FLUORIDE OF GLUCINUM AND OF OXYFLUORIDE OF GLUCINUM.

By P. LEBEAU.

BERZELIUS showed that a fluoride of glucinum could be prepared by the action of hydrofluoric acid on hydrate of glucinum. He described this compound as a body soluble in water in all proportions, drying to a colourless transparent mass, remaining limpid up to $+60^\circ$, but losing its water at 100° and becoming milky; at a higher temperature the substance becomes puffed up, and half melts like alum. Heated to redness it loses a part of its hydrofluoric acid if the desiccation has not been complete, but the residue from the calcination still dissolves in water, giving a clear solution.

We have taken up the study of this reaction, and have endeavoured to determine whether the product obtained was really anhydrous fluoride of glucinum or not. To this end, pure hydrated glucina was dissolved in pure hydrofluoric acid quite free from silica, and the clear solution was evaporated on a water-bath in a platinum dish. The liquid becomes concentrated into a gummy mass, which we were able, however, to bring to a powdery form by constant stirring. This substance, dried as perfectly

as possible at 100°, is not anhydrous fluoride of glucinum: the product, which is besides very deliquescent, always retains water; its proportion of glucinum never exceeds 17 per cent.

We next calcined the residue at different temperatures varying from 440° to 800°. In contact with air we observed the same phenomena as those described by Berzelius, viz., partial fusion, puffing, and complete solution in water.

Analysis showed that the substance heated to redness has a practically constant composition, corresponding to an oxyfluoride of the formula $5\text{GlF}_2, 2\text{GlO}$:—

	I.	II.	III.	Theory for $5\text{GlF}_2, 2\text{GlO}$.
Glucinum ..	22.01	23.31	22.90	22.26
Fluorine..	66.97	65.92	66.12	66.54
Oxygen (by dif.)	11.02	10.77	10.98	11.20

These analyses were made on three different samples:

The oxyfluoride is an almost transparent white body, completely soluble in water. The density at 15° is about 2.01.

Thus it is shown that anhydrous fluoride of glucinum cannot be obtained by the desiccation of the residue from the evaporation of a hydrated solution of fluoride of glucinum.

We next tried the evaporation and desiccation in a current of hydrofluoric acid gas. To effect this the substance, almost completely dried over the water-bath, was placed in a platinum boat which was in turn placed in a tube of the same metal; this tube was traversed by a regular current of hydrofluoric acid gas.

The temperature of the platinum tube was slowly and regularly raised to a bright red. After cooling, a melted transparent substance was found in the platinum boat closely resembling glass, but very deliquescent, the composition of which is that of a fluoride of glucinum, GlF_2 . There was also a ring of the same composition formed round the boat; it consisted of a melted vitreous part and a pulverulent deposit of small crystals. The analysis of this compound gave the following results:—

	Found.			Calculated for GlF_2 .
Glucinum ..	19.03	19.41	19.52	19.28
Fluorine ..	80.90	80.04	80.33	80.72
	99.93	99.45	99.85	100.00

We have identified this fluoride as being the same as that we prepared by the reaction of fluorine or of hydrofluoric acid gas on carbide of glucinum.

These methods for the formation of fluoride of glucinum only enabled us to prepare a small quantity of that substance; we therefore set to work to try and find a more practical method.

M. Camille Poulenc (*Ann. de Chim. et de Phys.*, Series 7, vol. ii.), in his researches on anhydrous fluorides, often made use of the calcination of ammoniacal fluorides. By this means he was enabled to obtain, starting from an ammoniacal salt, an anhydrous fluoride, sometimes amorphous and sometimes crystallised. We hoped to apply this method to the preparation of fluoride of glucinum. The double salt $\text{GlF}_2, 2(\text{NH}_4)\text{F}$, studied by Marignac (*Ann. de Chim. et de Phys.*, Series 4, vol. xxx., p. 47) is easily obtained crystallised, and can be dried perfectly. This salt, decomposed in a current of carbonic acid gas in a platinum vessel almost hermetically sealed, gave us an anhydrous fluoride of glucinum.

Properties of Anhydrous Fluoride of Glucinum.—Anhydrous fluoride of glucinum is found in the form of a vitreous transparent mass, or as a sublimate consisting of very deliquescent small crystals:— $D=2.1$ at 15°.

The fluoride melts in a similar manner to glass, taking a pasty condition, but it becomes very fluid towards 800°.

It is notably volatile at this temperature, giving a deposit of white crystals.

Fluoride of glucinum dissolves in water in all proportions. It is also slightly soluble in absolute alcohol, and very soluble in alcohol at 90°. By cooling an alcoholic solution to -23° a white mass of crystalline texture is formed; this melts very rapidly as soon as the temperature is raised. Etherised alcohol also acts as a solvent.

Most of the metalloids are without action on fluoride of glucinum. Oxygen transforms it into oxyfluoride; the vapour of sulphur has no effect whatever up to the temperature of the softening of glass.

Fluoride of glucinum is attacked by sulphuric acid. It forms an anhydrous sulphate and hydrofluoric acid. We have not succeeded in dissolving fluoride of glucinum in anhydrous hydrofluoric acid. By throwing the fluoride into the liquefied acid, there is no sensible rise in temperature. This experiment shows that the existence of an acid fluoride of glucinum is very improbable.

Fluoride of glucinum is reduced by sodium at a red heat, with the formation of fluoride of sodium, while metallic glucinum is set at liberty. The difficulty in fusing the fluoride and its hygroscopic properties cause this salt to be very inconvenient for the easy preparation of the pure metal. With potassium the reaction is much more energetic, and takes place with incandescence below 500°. Lithium behaves in an identical manner. Toward 650° magnesium decomposes fluoride of glucinum, under the same conditions. At the same temperature aluminium melts in contact with this compound without any reaction taking place.

To sum up, we have shown that the method of preparation given by Berzelius for obtaining a fluoride of glucinum leads to the formation of an oxyfluoride of practically constant composition, corresponding to the formula $5\text{GlF}_2, 2\text{GlO}$; and, further, we have succeeded in preparing the anhydrous fluoride GlF_2 , and have studied its principal properties.—*Comptes Rendus*, vol. cxvii., No. 20.

ON A BOROCARBIDE OF GLUCINUM.

By P. LEBEAU.

We have previously announced (*Comptes Rendus*, cxliii., p. 818) that boron had the power of reducing glucina at the temperature of the electric furnace; we now propose to study the compounds of boron and glucinum which may possibly be formed in this reduction.

Pure glucina, procured by the calcination of nitrate of glucinum, was mixed with a quantity of boron sufficient to remove all the oxygen and produce a boride. The actual proportions used were;—

Pure glucina ..	75 parts
Boron	45 "

These were thoroughly mixed, moistened with alcohol and compressed into the form of little cylinders, which were then dried by being left for some hours in an oven at 150°. One of these little cylinders was placed in a carbon boat, in a tube of the same material, and heated in the electric furnace. The heating lasted for seven or eight minutes with a current of 950 ampères and 45 volts. At the termination of the experiment the mass was found to be in a state of tranquil fusion. After cooling, a homogeneous material with a metallic appearance and a crystalline fracture was found in the boat.

This experiment was repeated on a much larger quantity of material in a carbon crucible. A button was formed at the bottom of the crucible, very hard, and of which the greater portion was metallic-looking and well-crystallised. In some places a white substance having undergone fusion could be distinguished; this was un-

reduced glucina. The metallic particles, collected with great care, were found to contain free glucina, and qualitative analysis showed that they were composed of carbon, boron, and glucinum. This substance is without action on water, even after several days' contact; the carbon is therefore not in the form of a carbide of glucinum easily decomposable by water, but, on the contrary, enters into a triple combination of carbon, glucinum, and boron.

The analysis of this compound was carried out in the following manner:—

1. *Estimation of the Boron and the Glucinum.*—A known weight of the material was attacked by nitric acid in the apparatus used by M. Moissan for estimating boron by Gooch's method. The boric acid entangled by the methylic alcohol was obtained directly by the increase in weight of a known quantity of quicklime. The residue remaining in the flask in which the attack took place was taken up with water, and thrown on a tared filter to get the weight of unreduced glucina which is insoluble under these conditions. The filtered liquor was precipitated with ammonia, and a small quantity of sulphide of ammonium added. The precipitate was weighed after calcination.

2. *Estimation of the Carbon.*—The carbon was estimated in the form of carbonic acid, the material being attacked by chromic acid in sulphuric solution. The arrangement of the apparatus was practically identical with that described by M. Carnot for the estimation of the carbon in irons and steels.

By deducting the quantity of unreduced glucina we obtained the following proportions between the weights of the carbon, the boron, and the glucinum combined:—

Carbon	27.99	28.34
Boron	29.18	39.12
Glucinum	32.83	32.54
		99.99

These figures have led us to give the formula $C_4B_6O_6Gl_6$, or $Bo_6C_3CGl_2$, to this carboboride of glucinum:—

	Calculated for $C_4B_6O_6Gl_6$.
Carbon	28.49
Boron	39.17
Glucinum	32.33

Borocarbide of glucinum occurs in brilliant crystals with a metallic lustre, having a density of about 2.4. It does not change in air at the ordinary temperature; heated to redness it oxidises superficially.

In a current of pure oxygen at about 700° the presence of carbonic acid may be detected, but the reaction stops rapidly, the layer of boric acid formed protecting the rest of the substance from any deeper oxidation.

Towards 45° borocarbide of glucinum burns in chlorine with a beautiful incandescence, chloride of boron and chloride of glucinum being produced, while a black residue remains consisting of amorphous carbon. Bromine gives an identical result; iodine is without action up to the softening point of glass. By operating in porcelain we noticed a very distinct reaction, with the production of iodide of glucinum and iodide of boron. In the vapour of sulphur, at a red heat, the attack takes place superficially; the residue when moistened with water gives off sulphuretted hydrogen.

The gaseous hydracids also react on the borocarbide of glucinum. The mineral acids, and particularly nitric acid, dissolve it rapidly.

We have not been able, in the reduction of glucina by boron in the electric furnace, to obtain any compound free from carbon. The reduction takes place at a very high temperature, and it was impossible for us to protect the matter from the vapours of carbon.—*Comptes Rendus*, vol. cxxvi., No. 19.

ON THE ABSORPTION OF NITRIC OXIDE BY FERROUS SALTS.

By V. THOMAS.

In a preceding paper, I showed that all ferrous salts, in whatever solution, absorbed nitric oxide, but that this absorption was more or less great according to the solvent used. I will here show, by referring to numerous experiments, that this absorption is not proportional to the minimum weight of iron contained in the solution, but that it is a function of the nature of the salt.

Absorption of Binoxide of Nitrogen by Ferrous Bromide. Solvent used: Water.

The absorptions were determined by weight and by volume.

Absorption by Weight.—A certain weight of bromide was dissolved in a quantity of boiled water, variable according to experience. To the solution iron filings were added, which, by prolonged boiling, collected all traces of the per-salt which might have been formed during the solution. This solution should be made in a flat-bottomed flask. In this flask a tube reaching below the surface of the liquid serves to convey a current of inert gas (CO_2 or H for preference). Another tube serves for the escape; this tube should be drawn out before the blowpipe, and should enter the lateral tube of a bulb gas-washer. This washer should be carefully tared. It is filled with the ferrous solution, taking care to exclude all air. For this purpose the lower end of the tube leading the gas into the flat-bottomed flask was kept above the surface of the liquid, while the other end was plunged into the solution. After having completely driven the air out of the apparatus, the binoxide of nitrogen was slowly passed through. This was partially absorbed, while the excess, carrying with it a little watery vapour, was passed through U-tubes filled with pumice-stone soaked in sulphuric acid and previously tared.

We could thus easily deduce the quantity of gas absorbed.

The contents of the washer were then collected, and on a known portion of the solution the iron was estimated in the state of oxide; this easily gives the concentration of the liquor.

Experiments below 10°. Temperature 6°.

I. Weight of iron dissolved	0.3795
Concentration of the liquor	0.75 per cent
Absorption	0.1360

II. Weight of iron dissolved	0.4970
Concentration of the liquor	0.6 per cent
Absorption	0.1740

Experiments above 10°. Temperature 10–12°.

I. Weight of iron dissolved	1.4846
Concentration of the liquor	2.5 per cent
Absorption	0.4045

II. Weight of iron dissolved	0.6230
Concentration of the liquor	1.25 per cent
Absorption	0.1665

If we calculate the percentage of iron into NO we get—

	I.	II.
Below 10°	35.1	35.0
Above 10°	27.2	26.6

Absorption in Volumes.—The method of operating has been described in a preceding note. We here give the results of the experiments. (See Table A).

TABLE A.

I. 2 c.c. of a solution containing	5.978 grms. Fe per 100 c.c. absorbed	30 c.c. NO at	6°
II. 9.1 " "	5.978 " "	27 " "	10°
III. 2.8 " "	5.978 " "	53 " "	-5°
IV. 2.8 " "	5.978 " "	45 " "	6°
V. 3.5 " "	5.978 " "	38.5 " "	11°
VI. 3.7 " "	5.978 " "	47 " "	8°
VII. 2.3 " "	5.978 " "	25 " "	8°
VIII. 4.1 " "	5.978 " "	65 " "	2°
IX. 3.5 " "	5.978 " "	38.9 " "	8°
X. 2.9 " "	5.978 " "	29.9 " "	10°
XI. 3 " "	5.978 " "	29.5 " "	10°
XII. 2 " "	5.978 " "	36.5 " "	0°
XIII. 1.5 " "	5.978 " "	25.4 " "	4°
XIV. 3.5 " "	5.978 " "	22.7 " "	20.5°
XV. 3.5 " "	5.978 " "	50.7 " "	4°
XVI. 2.5 " "	5.978 " "	41 " "	7°

TABLE B.

I. 11.2 c.c. of a solution containing	0.789 grm. Fe per 100 c.c. absorbed	21 c.c. NO at	17°
II. 16.5 " "	0.789 " "	28 " "	18°
III. 11.3 " "	0.789 " "	24.5 " "	13°
IV. 8 " "	3.110 grms. " "	52 " "	16°
V. 4.8 " "	3.110 " "	47 " "	12°
VI. 8 " "	3.110 " "	54 " "	15°
VII. 5 " "	3.110 " "	44 " "	14°

TABLE C.

I. 12.2 c.c. of a solution containing	3.171 grms. Fe per 100 c.c. absorbed	58 c.c. NO at	16°
II. 9.6 " "	3.171 " "	45 " "	13°
III. 8.5 " "	2.926 " "	36 " "	-3°
IV. 5.8 " "	2.926 " "	19 " "	18°
V. 8.3 " "	2.926 " "	37 " "	13°

TABLE D.

I. 8 c.c. of a solution containing	1.925 grm. Fe per 100 c.c. absorbed	23 c.c. NO at	15°
II. 9.1 " "	1.925 " "	26 " "	21°
III. 9 " "	1.925 " "	27 " "	12°

By examining the different proportions of NO present per cent of iron we get the following averages:—

Between -5° and 0° ..	41.73 per cent
" 0° " 7° ..	35.87 "
" 8° " 11° ..	24.90 "
At 20.5° ..	15.43 "

From these numerous determinations we can deduce that ferrous bromide does not exactly follow M. Gay's law; especially as, while with ferrous chloride the proportion of binoxide of nitrogen is 34.78 per 100 of iron at 12°, it is necessary, in order to get a similar absorption with bromide, to work at a temperature between 0° and 7°; and while towards 20° the chloride absorbs a quantity of gas corresponding to 26 per cent of the weight of iron, the bromide absorbs only 15 per cent of binoxide of nitrogen. When using an alcoholic solution, the results obtained are quite comparable.

Solvent used: Absolute Alcohol.—The absorptions were determined by volume (see Table B). The figures represent the following proportions of NO present:—

I. At 17° ..	28.33
II. " 18° ..	25.59
III. " 13° ..	35.52
IV. " 16° ..	28.02
V. " 12° ..	42.24
VI. " 15° ..	29.06
VII. " 14° ..	37.94

The same experiments made with the chloride at about 17° give a figure higher than 50 per cent of gas (per cent of iron). We must therefore conclude that the absorption is less with the bromide than with the chloride. Experiments made with the iodide lead to entirely analogous results.

Absorption of Binoxide of Nitrogen by Ferrous Iodide.

Solvent used: Water.—The absorptions were determined by volume (see Table C). The proportions of nitric oxide present per cent of iron are:—

I. At 16° ..	18.29
II. " 13° ..	19.28
III. " -3° ..	20.62
IV. " 18° ..	17.41
V. " 13° ..	20.43

It is as well to remark here that temperature has but little influence on gaseous absorption.

Solvent used: Alcohol.—(See Table D). The proportions of NO present in percentages of iron are:—

I. At 15° ..	20.00
II. " 21° ..	19.95
III. " 12° ..	20.89

The same remark again applies here. In a future paper I propose developing some conclusions resulting from these experiments.—*Bull. Soc. Chim., Series 3, vol. xix.-xx., No. 18.*

A DETERMINATION OF THE
ATOMIC WEIGHT OF PRASEODYMIUM AND OF
NEODYMIUM.*

By HARRY C. JONES.

(Concluded from p. 282).

THE method first employed consisted in dissolving the superoxide in sulphuric acid, evaporating the solution of the sulphate to dryness, and heating it in a platinum vessel to a temperature above the boiling-point of sulphuric acid. The sulphate was then weighed, dissolved in water to form a very dilute solution, strongly acidified with hydrochloric acid, and the sulphuric acid precipitated by means of barium chloride. It was hoped that by using a very dilute solution, strongly acidified, it would be possible to precipitate all of the sulphuric acid as the barium salt. Several determinations were made, but the results obtained were so discordant as to be entirely valueless. The presence of praseodymium was shown in the precipitate, even after it had been boiled with excess of barium chloride for a considerable time. This fact had been pointed out by Marignac (*loc. cit.*), but it was hoped that by working in very dilute solutions, which had been strongly acidified, the difficulty could be overcome. It could not, and the method had to be abandoned as entirely useless for the purpose of an atomic weight determination.

The method adopted consisted in reducing the superoxide to the sesquioxide in an atmosphere of hydrogen, and converting the sesquioxide into the sulphate. The details of the process are as follows:—

A weighed amount of the superoxide was placed in a porcelain boat, and this within a hard-glass tube, which rested upon a combustion furnace. A stream of hydrogen gas, which had been washed in a solution of sodium plumbate, to remove hydrogen sulphide, and in silver nitrate to remove arsine, was dried over calcium chloride, and passed through the tube. The superoxide was easily reduced to the sesquioxide on heating. The sesquioxide was allowed to become perfectly cold in the current of hydrogen, and boat and contents were then removed from the tube and weighed. The boat containing the sesquioxide was then returned to the glass tube and re-heated in an atmosphere of hydrogen. In no case was there any change in weight caused by the second heating.

A platinum crucible was placed within a ground-glass stoppered weighing-tube, which was then closed and weighed. The sesquioxide was poured from the boat into the crucible, and the whole was re-weighed. This gave the weight of the sesquioxide used in a determination.

A considerable excess of sulphuric acid, of medium concentration, was added to the crucible. An equal volume of the sulphuric acid used was placed in a weighed platinum crucible, evaporated to dryness, and heated exactly as in carrying out a determination, to see whether there was any appreciable residue left behind. In no case did the weight of the crucible increase more than 0.00005 gm., which was therefore negligible. The platinum crucible, containing the oxide and sulphuric acid, was placed in an air-bath constructed of thick sheet copper, and closed at the bottom by a piece of very thick sheet copper. The sides of the bath were thickly covered with asbestos paper. The platinum crucible rested upon a porcelain triangle, about an inch from the bottom of the air-bath. Heat was applied to the bath, very gently at first, to avoid all spattering, and then increased, until the solution of the sulphate had evaporated nearly to dryness. Then the bath was heated more strongly, and finally raised to a temperature above the boiling-point of sulphuric acid. The sulphate was maintained at this tem-

perature for some hours, and then weighed. It was re-heated and re-weighed, and this continued until constant weight was reached. To determine whether only the normal sulphate was present, purified ammonium carbonate was added, and the crucible and contents again heated above the boiling-point of sulphuric acid. There was no change in the weight of the sulphate, showing that only the normal sulphate of praseodymium was present. The sulphate thus prepared was perfectly soluble in water. I was completely unable to verify the observation of Bailey (*Y. Chem. Soc.*, li., 676), who stated that "the sulphate was made into a paste with sulphuric acid and heated at 360°. . . . An approach to a constant weight was manifest, but variations amounting to a m.grm. or more occurred, and with each successive increase of temperature the loss was accelerated. No range of temperature could be found within which the salt remained perfectly constant."

I found that the weight of the sulphate remained perfectly constant through a considerable range of temperature, provided the temperature to which it had been heated was sufficient to remove all the excess of sulphuric acid and decompose any acid sulphate. It is possible that Bailey did not heat the sulphate, or in his case the mixture of praseodymium and neodymium sulphates, to a temperature sufficiently high to leave only the normal sulphate. This is the only way I can account for the result which he obtained.

The crucible containing the sulphate was removed from the air-bath as quickly as possible and placed in the weighing-tube. This was closed and placed in a desiccator over phosphorus pentoxide and allowed to cool. The sulphate was then weighed in the closed tube, which is necessary, since it absorbs moisture rapidly from the air.

The Results.

	Pr ₂ O ₃ .	Pr ₂ (SO ₄) ₃ .	3(SO ₃) ₂ .	{ O=16 S=32.07 } At. wt. Pr.	{ O=15.88 S=31.83 } At. wt. Pr.
1	0.5250	0.9085	0.3835	140.42	139.37
2	0.6436	1.1135	0.4699	140.50	139.45
3	0.7967	1.3788	0.5821	140.38	139.33
4	0.7522	1.3018	0.5406	140.38	139.33
5	0.7788	1.3473	0.5685	140.53	139.48
6	0.6458	1.1172	0.4714	140.54	139.49
7	0.6972	1.2062	0.5090	140.51	139.46
8	0.7204	1.2464	0.5260	140.49	139.44
9	0.8665	1.4990	0.6325	140.54	139.49
10	0.6717	1.1624	0.4907	140.40	139.35
11	0.7439	1.2873	0.5434	140.42	139.37
12	0.6487	1.1224	0.4737	140.47	139.42
	8.4905	14.6908	6.2003	140.46	139.41

The average of the individual determinations is 140.46 when oxygen = 16, or 139.41 when oxygen = 15.88. The difference between the highest and lowest value is 0.16. This series represents all of the determinations which were made, with the exception of two, which were lost by accident and never completed.

The atomic weight of praseodymium, as calculated from the total oxide used, and total sulphate formed, is 140.47 when O=16, or 139.41 when O=15.88.

The sesquioxide, prepared by reducing the superoxide in a current of hydrogen, always liberates a small amount of gas when dissolved in sulphuric acid. This is probably occluded hydrogen. The amount of gas thus set free was determined approximately, and found to be so small that it could be disregarded.

The atomic weight of praseodymium, as here determined, may be taken as 140.45 when oxygen = 16, or 139.4 when oxygen = 15.88. This result lays no claim to very great accuracy, because of the apparent impossibility of obtaining an absolutely pure praseodymium compound. However, considering the result of the careful spectroscopic analysis which was made of the material

* From the *American Chemical Journal*, vol. xx., No. 5.

which I employed, and all the errors which could possibly have entered into the determinations, I see no reason to think that the result can differ from the true atomic weight of praseodymium by more than 0.2 of a unit.

The Atomic Weight of Neodymium.

Somewhat more than 2 kilograms. of the double nitrate of neodymium and ammonium were furnished me by Mr. Shapleigh. This was analysed spectroscopically, and found to contain praseodymium and also a trace of lanthanum. The substance was further purified by fractional crystallisation, as described for praseodymium. The entire material was dissolved in water, to which a little nitric acid had been added and evaporated, until, on cooling, about 1/15 to 1/20 crystallised out. A considerable volume of nitric acid was then added, and the larger part of the double salt allowed to separate on further concentrating the solution. The mother-liquor was then carefully filtered from the crystals. The first and third fractions were discarded, and the second preserved for further treatment. The middle fraction was then treated exactly as the original substance had been, was separated into three fractions, and the middle one preserved and again treated in the same manner. This process was repeated twenty-seven times.

The middle fraction of the last crystallisation was dissolved in water, strongly acidified with nitric acid, and poured into a dilute solution of oxalic acid, in the same manner as described under praseodymium. This would separate such common impurities as iron, calcium, &c. The oxalate was washed with great thoroughness, dried, and decomposed in a platinum crucible over the blast-lamp. The oxide of neodymium, thus prepared, was again analysed spectroscopically. It was found to contain praseodymium and a trace of lanthanum. The same method was employed to separate the trace of lanthanum as in the case of the praseodymium salt.

The praseodymium which remained in the double salt of neodymium could not be separated by any known method, and must therefore be determined. The amount present was ascertained with considerable accuracy by studying the intensity of the absorption bands, and comparing these with the absorption bands of the pure praseodymium salt of known concentration. The exact method is that described under praseodymium. It was found that the double nitrate of neodymium and ammonium contained 1.6 per cent of the double nitrate of praseodymium and ammonium.

After the lanthanum was separated the neodymium was again precipitated as the oxalate, with oxalic acid, in the presence of nitric acid, by the method already described. The oxalate was converted into the oxide by heating in a platinum crucible over the blast-lamp. The oxide thus obtained was analysed spectroscopically, special attention being given to the presence of thorium. The only impurity which could be detected was praseodymium, the amount of which had already been determined.

The oxide of neodymium, obtained from the oxalate, does not possess any property of a superoxide as such. It dissolves in sulphuric acid without the evolution of oxygen. When heated in a current of hydrogen exactly as the oxide of praseodymium obtained from the oxalate, it does not change in weight. When hot, the oxide has a marked bluish colour, which, however, disappears on cooling. It will thus be seen that the substance which I employed in determining the atomic weight of neodymium had all the properties described by Von Welsbach (*Monatsh. Chem.*, vi., 489), as characteristic of the sesquioxide of neodymium.

The method employed in determining the atomic weight of neodymium was the same, in general, as that used in the case of praseodymium. After it had been found that the oxide obtained from the oxalate did not change in weight when heated in a current of hydrogen, this part of the process was abandoned. The oxide was,

however, heated in the platinum crucible just before weighing, and allowed to cool in a desiccator over phosphorus pentoxide, to remove every trace of moisture. The method of synthesising the sulphate from the oxide, and heating and weighing the sulphate, was the same as employed with praseodymium.

The Results.

	Nd ₂ O ₃ .	Nd ₂ (SO ₄) ₃ .	3SO ₃ .	{ O=16 } { S=32.07 }		{ O=15.88 } { S=31.83 }	
				At. wt. Nd.	At. wt. Nd.	At. wt. Nd.	At. wt. Nd.
1	0.8910	1.5296	0.6386	143.58	142.50		
2	0.7880	1.3530	0.5650	143.51	142.43		
3	0.9034	1.5509	0.6475	143.57	142.49		
4	0.7668	1.3166	0.5498	143.51	142.43		
5	0.8908	1.5296	0.6388	143.49	142.41		
6	0.8848	1.5194	0.6346	143.46	142.38		
7	0.8681	1.4903	0.6222	143.57	142.49		
8	0.8216	1.4103	0.5887	143.62	142.54		
9	0.8531	1.4646	0.6115	143.56	142.48		
10	0.8711	1.4957	0.6246	143.50	142.43		
11	0.8932	1.5332	0.6400	143.62	142.54		
12	0.8893	1.5268	0.6375	143.55	142.47		
				10.3212	17.7200	7.3988	143.55 142.47

The average of the twelve determinations is 143.55 when oxygen = 16, or 142.47 when oxygen = 15.88. The difference between the highest and lowest value is 0.16. Three determinations were lost by accident. The atomic weight of neodymium, calculated from the total amount of oxide used and sulphate formed, is 143.55 when oxygen = 16, or 142.47 when oxygen = 15.88.

The value of the atomic weight of neodymium thus found must be corrected for the presence of praseodymium, which has an atomic weight about three units lower. The final value, which, from my work, I should assign to the atomic weight of neodymium, is 143.6 when oxygen = 16, or 142.52 when oxygen = 15.88. I think this value can be regarded as accurate to within about the same limits as indicated in the case of praseodymium.

The oxide of neodymium, prepared as above described, liberated a small amount of gas, presumably air, when dissolved in sulphuric acid. But since this work lays no claim to the last degree of accuracy, due chiefly to the impossibility of effecting complete separations of the rare elements, such corrections were found to be less than the necessary experimental error.

The values which I obtained for the atomic weights of praseodymium and of neodymium differ widely from those given by Von Welsbach (*Monatsh. Chem.*, vi., 490). It is impossible to determine from the paper of Von Welsbach what he regarded as the atomic weight of oxygen; but since no statement is made, the presumption is in favour of 16. The following comparison will show how widely the two results differ:—

	Von Welsbach.	My results.
Pr	143.6	140.45
Nd	140.8	143.6

There is a discrepancy of about three units in the case of each element. Indeed, if my values were reversed for the two elements, they would not differ widely from those of Von Welsbach.

It is not possible to account satisfactorily for the difference between my results and those of Von Welsbach, since the latter has given no details pertaining to his determinations. The fact that if either set of results were interchanged for the two elements they would agree with the other, suggests that possibly this discrepancy arose in a typographical error in the paper of Von Welsbach, in which his values are recorded as tentative, and in less than a line.

THE ACTION OF CARBON DIOXIDE ON
SOLUBLE BORATES.*

By LOUIS CLEVELAND JONES.

In a process for the separation and estimation of boric acid devised by Morse and Burton (*Am. Chem. Journ.*, x., 154), the liberation of carbonic, silicic, and boric acids from a mixture of inorganic salts is effected by the action of sulphuric acid, using tropæolin OO as an indicator of acidity. In the solution thus prepared, containing in free condition only carbonic, silicic, and boric acids, the silicic acid is dehydrated and made insoluble by anhydrous copper sulphate. The boric acid is then extracted with absolute alcohol. To this alcoholic solution of boric acid a known amount of barium hydroxide solution is added in excess over that required to form a barium metaborate, BaB_2O_4 . Carbon dioxide is then passed into the solution in accordance with the hypothesis that the excess only of barium is acted upon. The aqueous mixture of barium metaborate and barium carbonate is evaporated, and the residue is heated to a constant weight over a triple burner. From the following proportion the boric acid present may be calculated:—

The molecular weight of boric acid — the molecular weight of carbon dioxide : the molecular weight of boric acid = the total weight found — the theoretical weight of barium as carbonate : the weight of boric acid present.

It is obvious, inasmuch as the difference between the calculated weight of the barium as carbonate and the actual weight of the residue is multiplied nearly three times to get the boric oxide, that the actual error of the process, whatever it may be, is magnified three-fold by the method of computation.

I have made a study of this method applied to pure boric acid, but have been unable to obtain results similar to those of Morse and Burton.

For this investigation boric acid of standard strength was made by dissolving in a given amount of water a known weight of anhydrous boric oxide, prepared by igniting over a blast lamp boric acid several times recrystallised and washed. A solution of barium hydroxide was filtered free from carbonate and then standardised by precipitation as carbonate and also by the Phelps method with iodine (*Am. Chem. Journ.*, ii., 70). To a measured amount of the boric acid solution an excess of barium hydroxide was added, carbon dioxide passed, and the whole evaporated and by successive ignitions brought to a constant weight. Below are tabulated some of the results (see Table, next column).

Plainly the results vary with the degree of the ignition. At the outset the residue may or may not weigh more than the theory requires for the known amounts of barium hydroxide and boric acid taken upon the assumption that the residue is barium metaborate and barium carbonate. This is obviously natural, if the carbon dioxide acts upon the barium borate as well as upon the excess of barium hydroxide; for, it is to be expected that in the evaporation more or less of the free boric acid will volatilise, and that in the subsequent ignition the boric acid remaining will tend to re-combine more or less completely, replacing carbon dioxide. If the boric acid present were to re-combine completely with the barium carbonate to form a metaborate, the final result would always be low by just the amount of free boric acid volatilised in the process of evaporation and ignition. The evidence of an experiment, however, in which 0.25 gm. of previously prepared barium metaborate was fused in contact with 0.5 gm. of barium carbonate, resulting in a loss of 0.0871 gm., goes to show that the metaborate and carbonate of barium interact still further to liberate carbon dioxide.

	Ba(OH)_2 taken. Calculated as BaCO_3 . Grm.	B_2O_3 taken. Grm.	Weight of residue after successive ignitions. Grm.	B_2O_3 found. Grm.	Error. Grm.
I.	0.9391	0.2200	1st wt., 0.9860 2nd „ 0.9786	0.1263 0.1063	-0.0937 -0.1137
II.	0.9318	0.1295	1st „ 0.9605 2nd „ 0.9558 3rd „ 0.9510	0.0744 0.0646 0.0517	-0.0523 -0.0649 -0.0778
III.	0.9253	0.2192	1st „ 1.0357 2nd „ 1.0248 3rd „ 1.0149 4th „ 1.0064 5th „ 0.9975 6th „ 0.9855	0.2972 0.2679 0.2412 0.2183 0.1944 0.1621	+0.0780 +0.0487 +0.0220 -0.0009 -0.0248 -0.0671
IV.	0.7301	0.0810	1st „ 0.8017 2nd „ 0.7777 3rd „ 0.7642 4th „ 0.7582 5th „ 0.7517 6th „ 0.7482 7th „ 0.7447 8th „ 0.7427 9th „ 0.7422 10th „ 0.7407	0.1927 0.1281 0.0918 0.0757 0.0582 0.0487 0.0393 0.0339 0.0326 0.0285	+0.1117 +0.0471 +0.0108 -0.0053 -0.0228 -0.0323 -0.0417 -0.0471 -0.0484 -0.0525

These results were so surprising in the light of the experience of Morse and Burton that the question of the possibility of breaking up by carbon dioxide the barium metaborate already formed was put to the test directly. A known amount of barium hydroxide was taken in solution and to it added an amount of boric acid very little in excess of that theoretically necessary to form the barium metaborate. The solution was evaporated to dryness and the residue ignited. The weight obtained proved to be 0.0008 gm. less than the sum of the barium and boric oxides taken, doubtless because the slight excess of boric acid was somewhat volatile in the evaporation. The mass, presumably barium metaborate, was now dissolved as completely as possible in hot water, carbon dioxide was passed through the solution, the whole was evaporated, and the residue ignited and weighed. The increase in the weight showed that carbon dioxide had been absorbed, while a corresponding amount of boric acid had not volatilised.

	Ba(OH)_2 taken. Calculated as BaCO_3 . Grm.	B_2O_3 taken. Grm.	Residue after ignition. Grm.	Residue after CO_2 treatment and ignition. Grm.
V.	0.8377	0.2990	0.9491	-0.9771

After passing in carbon dioxide and igniting, the increase in weight was 0.0280 gm., representing the gas absorbed less the boric acid volatilised.

It was plain that barium metaborate is decomposed in solution by carbon dioxide. The possibility remained, however, that the action of carbon dioxide might be so regulated as to leave the metaborate practically unattacked. In experiment VI., therefore, carbon dioxide was passed above the stirred solution until no further precipitate formed upon the surface, the barium present being in excess of that required to form a metaborate.

	Ba(OH)_2 taken. Calculated as BaCO_3 . Grm.	B_2O_3 taken. Grm.	Weight of residue after ignition. Grm.	B_2O_3 found. Grm.	Error. Grm.
VI.	0.7094	0.2070	0.7710	0.1658	-0.0412

The variation of this result from the theory shows that under these conditions the metaborate is not unaffected by carbon dioxide, the loss being due, of course, to the escape of boric acid.

An attempt was now made therefore to gauge the amount of the carbon dioxide introduced by means of an indicator. In Experiment VI. phenolphthalein was added to the solution of boric acid containing an excess

* Contribution from the Kent Chemical Laboratory of Yale University. From the *American Journal of Science*, vol. v., p. 442.

of barium hydroxide, and the current of gas was stopped when the colour of the indicator disappeared.

Ba(OH) ₂ taken. Calculated as BaCO ₃ .		Ba ₂ O ₃ taken.	Weight of residue after ignition.	Ba ₂ O ₃ found.	Error.
Gm.	Gm.	Gm.	Gm.	Gm.	Gm.
VII. 0.6073	0.1573	1st wt., 0.6820	0.2011	+0.0438	
		2nd „ 0.6783	0.1911	+0.0338	
		3rd „ 0.6720	0.1742	+0.0169	
		4th „ 0.6700	0.1688	+0.0115	
		5th „ 0.6655	0.1567	-0.0006	
		6th „ 0.6630	0.1499	-0.0074	
		7th „ 0.6609	0.1443	-0.0130	

This result is manifestly an improvement over those obtained without the careful restriction of the supply of carbon dioxide. A similar experiment, differing only in the single point that the carbon dioxide was made to act upon a boiling solution, resulted in like manner.

In the light of these observations it is plain that a sufficiently prolonged action of carbon dioxide should result in the displacement of all the boric acid if that acid can be removed from the field of action as fast as it is liberated. Experiments were made which clearly demonstrate the truth of this hypothesis. A small side-necked flask was charged with a solution of boric acid (0.1143 gm.) and barium hydroxide (0.3227 gm.) in proportion to form the metaborate. The mass was brought nearly to dryness by distillation and methyl alcohol (15 c.m.) added. Through this flask, in which the alcohol was kept boiling by a Bunsen burner, was passed the vapour of methyl alcohol, while carbon dioxide, purified by a neutral solution of silver nitrate, bubbled continually through the entire system. The methyl alcohol vapour coming from the side-neck flask was kept lighted by contact with the flame of a Bunsen burner and the distillation continued for two hours—until the flame showed not the slightest tinge of green. The residue in the flask originally containing barium metaborate was brought to dryness and tested for boric acid. Only a trace was found, and this was thought to be due to inclusion by the insoluble barium carbonate. In a similar experiment, in which borax was used instead of barium borate, no trace of boric acid was found in the residue of sodium carbonate, either by turmeric or the flame test, while the distillation was continued only one-half the time of the preceding experiment. This result is quite in harmony with the views of P. Georgevic (*Journ. Prakt. Chim.*, xxxviii., 118) to the effect that the large absorption of carbon dioxide in solutions of borax indicates that the boric acid is displaced from its union with the base.

Obviously, the division of the base between boric acid and carbonic acid falls under the principle of mass action, and if the boric acid is taken in sufficient excess over the barium hydroxide, the action of the carbonic acid should be inappreciable. This idea is sustained by an experiment in which about 1 gm. of boric acid was dissolved with 0.15 gm. of barium hydroxide. No precipitate could be obtained by passing carbon dioxide and boiling. In fact, in the French process of borax manufacture just this action of an excess of boric acid upon a boiling solution of sodium carbonate is used. On the other hand, to prevent the formation of a carbonate this excess of boric acid must be considerable if the action of carbon dioxide is prolonged. Thus, in an experiment in which a current of carbon dioxide was passed into a solution containing 0.1219 gm. of boric anhydride and one-half the amount of barium needed to form a metaborate, the solution (60 c.m.) deposited on boiling 90 per cent of the barium present in the form of the carbonate.

In view of the facts which I have described, it is difficult to see under what conditions Morse and Burton prevented the excessive action of carbon dioxide and obtained in their analytical method the excellent results which they record.

I wish to express my thanks to Professor Gooch, whose advice has been constantly sought and freely given.

NEW CONSTITUENTS OF THE ATMOSPHERE.

At the meeting of the Royal Society on June 9th, Professor Ramsay and Mr. Morris Travers described their identification of a new constituent in atmospheric air, which they designated krypton. At the same time they intimated that, quite recently, during their investigations, they had found indications of another gas which afforded every promise of being also a novelty. They have now completed a preliminary examination of this constituent, and gave an outline of its nature and history at the meeting of the Royal Society on June 16th.

The discovery has been made in the course of a series of researches into the nature of argon which has occupied them for many months at the laboratory in University College, and in which they have been greatly aided by having at their disposal the apparatus devised by Dr. Hampson for liquefying air in large quantities. In view of these researches, they had provided themselves with a large quantity of the gas argon, the discovery of which in atmospheric air was announced by Lord Rayleigh and Professor Ramsay in 1895—altogether, some 18 litres, or, in other words, about 15 quarts. Already the latter investigator, in conjunction with Dr. Norman Collie, had attempted to separate the argon—in which they already suspected the presence of a second constituent in small quantities—into a lighter and heavier section, by diffusion, but, though they met with partial success, the difference in density between the two parts was so slight that they hesitated to draw the conclusion that the argon with which they were dealing was in reality a composite substance. But the experiments with helium which, as described at the Toronto Meeting of the British Association, were carried on by Messrs. Ramsay and Travers, in hope of finding a new gas intermediate between it and argon, led them to the conclusion that the greatest difficulty attended the attempt to separate a very small portion of a heavy gas from a large quantity of a light one by the means of diffusion; so they determined, since they had the use of Dr. Hampson's apparatus, to attack the problem in quite another way.

A quantity of argon was liquefied. It formed a colourless fluid, but at the same time a considerable quantity of a solid substance was observed to separate, partially round the sides of the tube and partially below the surface of the liquid. But this more conspicuous material was not all. A gas also remained, which was at once removed for further examination. After that they turned their attention to the frozen material, which was also separated by processes which they described. The light gas, which was the immediate subject of their investigation, was first examined. The spectrum was characterised by a number of bright red lines, one being particularly brilliant, and a yellow one of similar intensity. Green and blue lines were numerous, but inconspicuous. The yellow line is as intense as those in sodium, helium, and krypton, but is not identical with them. Next, an effort was made to ascertain the density of the gas. The results of their experiment led the investigators to the conclusion that they had not as yet obtained it in a perfectly pure condition. A sample, taken off at an early stage, gave a density of 17.2; at a later one, however, it was reduced to 14.67. But this is still too high if the new gas conforms to the Periodic Law—for, in that case, it should not exceed 11. It may, however, be lowered by further purification. But these tests, and sundry others, suggest that, even if not yet obtained in an absolutely pure condition, this gas is a new element, for which, accordingly, the name *Neon* is proposed. They next turned their attention to the solid—the material frozen out of argon. Its spectrum proved to be very complex, but totally different from that of argon, and its behaviour at low temperatures was markedly different, though its density was practically identical. It was proved to consist of a single element. This substance, accordingly, which was separated by freezing out of argon,

must be a distinct elementary body, though in some respects in close relationship with it, so they propose to name it *Metargon*. In fact, as the investigators observe, it occupies the same position in regard to argon that nickel does to cobalt, having the same atomic weight, yet different properties.

In this account no mention is made of krypton, the discovery of which was announced last week. Naturally it will be asked why the investigators did not again come across it. They attribute its absence to one, or possibly both, of two causes. In order to prepare it they would have required a sample not less than sixty thousand times the volume of that which they used in the above-mentioned experiments, and, besides this, metargon is a solid at the temperature of boiling air, while krypton is probably volatile. Moreover, the air from which they originally obtained the latter gas had been filtered, and thus separated from metargon. Spectroscopic investigation, which has been undertaken by Mr. Baly, will have to be carried further, and, if possible, applied to more highly purified samples, before these new elements can be completely known. The results of such investigation may account, as was intimated in the course of the discussion, for certain peculiarities in the spectrum of argon which, when this gas was first separated, led more than one experienced investigator to doubt whether it was in reality a single element, and not a compound of at least two. This question will be settled by a careful comparison of the spectra of argon and metargon, when it will be ascertained whether the lines in the former, which had caused doubt, disappear when a perfectly pure sample of it is examined, but are found to occur in the spectrum of metargon. Messrs. Ramsay and Travers expressed themselves fully sensible of the importance of following up these hints, and stated that they had prepared a quantity of thoroughly pure argon, which they trusted would be before long subjected to spectroscopic examination, while the spectra of krypton and neon would be watched at every stage as they were obtained, and especially the latter, in a more and more pure condition. One of the greatest difficulties in such investigations is the chemical inertness of these new gases. Still, the resources of chemical ingenuity are not exhausted.

The most interesting point which, in the case of neon, awaits further examination, is the determination of its density and atomic weight. As we have already said, a gas with a density of 10, or, at any rate, not more than 11, should exist, if the Periodic Law of Mendeleeff hold good, in the interval between argon and helium. It was this gas for which Professor Ramsay was hunting during the earlier part of last year. It was this which, as he described to the Chemical Section of the British Association at Toronto, he failed to obtain by methods of diffusion, though his experiments were numerous and varied. But this gas neon seems very likely to be the missing link; its density has already been reduced by processes of purification from 17 to 14 (in round numbers), and the investigators are convinced that a further lowering will be made as they continue their experiments. It is then highly probable, we might venture to say almost certain, that neon will ultimately prove to be the gas which managed to elude them on the first search. The moral is the homely one, "Try again" but by some other way.

Not the least suggestive part of the remarkable discoveries which have signalled the closing month of the Royal Society's Session is the way in which one discovery has led on to another, and that which at first seemed only a feat of the laboratory has turned out to be an agent of research of the highest scientific value. When Picot and Cailletet, rather more than twenty years ago, first liquefied air, they had probably little idea that they had done more than ascertain a fact of great scientific interest. But they had in reality laid the foundation-stone of a great superstructure of most important research. This idea was followed up by other investigators in various countries of Europe, including our own; their apparatus

was improved, or new was devised, and now liquid air can be produced in large quantities, and at a comparatively moderate price. The researches of Professor Dewar at the Royal Institution, of Dr. Hampson in his own laboratory, and of Professor Ramsay, with Dr. Collie and Mr. Travers, have been to a very great extent dependent on this liquid. It might be said to have swept away a barrier during the last very few years, and to have revealed a widening field of scientific knowledge. Not only air, but such liquid gases as oxygen, nitrogen, and argon will all prove helpful in further investigations, and it would be idle to attempt to predict the results which may follow from their employment. Certain it is that the interesting researches which have been described this month are not likely to be the last, for the laboratories in Albemarle Street and Gower Street will be sure to vie one with another in striving to find out some new thing in the air we breathe, the water of springs, and the minerals of the earth.—*The Standard*, June 17th, 1898.

NOTICES OF BOOKS.

Laboratory Experiments on the Class Reactions, and Identification of Organic Substances. By A. A. NOYES, Ph.D., and S. P. MULLIKEN, Ph.D. Second Edition, thoroughly Revised. Pp. 28. Easton, Pa.: Chemical Publishing Company. 1897.

THE primary object of the experiments herein described is to illustrate the characteristic reactions of organic substances, though the identification of unknown compounds is also a matter of great importance, and it is hoped by the authors that the student who intelligently follows the course of instruction here given will be able to devise methods for the detection and separation of other bodies not given in this volume.

The book is divided into three parts:—Part I. gives experiments illustrating the class reactions of organic compounds, such as distinguishing double- and triple-bonded from single-bonded compounds; distinguishing hydrocarbons of different series; halogen compounds of different types; saturated and aromatic hydrocarbons, and their halogen derivatives from other compounds; various reactions, &c.

Part II. illustrates the methods of detecting nitrogen, sulphur, and halogens in organic compounds; and Part III. deals with the identification and separation of unknown organic substances.

We have thus a very useful and clearly written book, which will be of great service to the student working on organic chemistry.

French Self-taught, with Phonetic Pronunciation. By C. A. THIMM, F.R.G.S. London: E. Marlborough and Co. Pp. 93.

ALTHOUGH there is a great deal that is useful in this phrase book, we cannot admit that the author's phonetic method of teaching French pronunciation is altogether satisfactory—there is a great deal too much "g" in it; many of the words, though curious-looking (in some cases almost grotesque), must be admitted to be correct; but we do object to "soovaung" for "souvent," "maing-tenaung" for "maintenant," "sepaundaung" for "cependant," &c. This is merely accentuating and encouraging what may be considered to be one of the chief faults in an ordinary Englishman's pronunciation of French, and one which shows imperfect teaching.

The general arrangement of the book and the choice of phrases and sentences are all that can be desired, and to anyone touring in France, with only a slight knowledge of the language, it will no doubt be of service.

Tables for Qualitative Chemical Analysis. ("Tafeln zur Qualitativen Chemischen Analyse"). By W. HAMPE. Fourth Edition, Revised and Enlarged. Clausthal. 1897.

THIS collection of Tables for Qualitative Analysis, of which there are twenty-one in all, may be divided under three heads:—Tables I. to IX. are on the principal reactions of bases and acids; Tables X. and XI. on the testing of substances in the dry way; and Tables XII. to XXI. on the systematic method of procedure in qualitative analysis.

Though not arranged in the groups we are most familiar with in England, these tables will be of great use to the student who understands German. It is always well to know of more than one method of arriving at a wished-for result.

CORRESPONDENCE.

THE NEW GAS.

To the Editor of the Chemical News.

SIR,—Your anonymous correspondent of last week has omitted to mention the name of Lord Rayleigh, in attributing to me the announcement that argon is the inert constituent of atmospheric air; and also the name of Mr. Morris Travers, in connection with his remarks on Krypton. I hasten to correct these errors, which, doubtless through inadvertence, have escaped your notice.—I am, &c.,

WILLIAM RAMSAY.

University College, London,
Gower Street, W.C., June 21, 1898.

THE NEW GASES.

To the Editor of the Chemical News.

SIR,—Your correspondent, "Suum Cuique," apparently cannot see that Prof. Ramsay had added anything "to what Cavendish proved."

If I have formed a correct conception of Cavendish's work, he proved that if air contained any other substance than oxygen, nitrogen, and carbon dioxide, it did not exceed a certain small amount; though he actually isolated other gases, he never knew for certain that he had done so, and his account of his experiments was in such terms as to lead others of his day to believe that air was composed of only oxygen, nitrogen, and carbon dioxide. In fact Cavendish, as an experimenter, was in advance of his time, and his marvellous work was not capable of being properly interpreted till much later.

The history of the discovery of argon, &c., seems to me to have been thus:—The real discoverer was Lord Rayleigh, who observed that the density of atmospheric nitrogen was greater than that of chemical nitrogen: as a physicist, he doubtless felt that it would be desirable to associate himself with a chemist, and therefore Rayleigh and Ramsay isolated the constituent of air, which Cavendish did not know that he had obtained. So far, to my mind, the credit has been chiefly Lord Rayleigh's.

I am not aware that it was ever claimed that argon was proved to be an entity, and I gathered that Rayleigh and Ramsay had the possible mixed nature of argon constantly before their minds.

Next, led by the research of Hildebrand, Ramsay accidentally discovered helium; from this time I have gathered, from Ramsay's work, the possible mixed nature of both helium and argon was strongly before him. He spent many months trying to split up helium, and succeeded in separating a little argon, &c., from it: now it seems that he is attacking argon in the same way, and

has already separated a gas which withstands the action of red-hot magnesium and calcium on one hand, and, on the other, sparking with oxygen in the presence of caustic soda; which is monatomic, which has a density greater than that assigned to argon, and which has a spectrum containing lines not belonging to any other body. On the whole of this evidence he concludes that there is a hitherto undescribed constituent of the atmosphere.

That Prof. Ramsay is fully alive to the possibility of other gases being present in the atmosphere, is shown by the fact that he and Mr. Travers have spent many months in the preparation for a search for another constituent. It is indeed chiefly in the careful work with the object of establishing the simple or mixed nature of helium, argon, &c., that the credit of Ramsay lies.

If it had not been for Rayleigh and Ramsay "Suum Cuique" would have probably lived in blissful ignorance of the classical experiment of Cavendish. As chemists have waited a century to learn "what Cavendish proved," cannot "Suum Cuique" wait a year or so, and give Prof. Ramsay time to do the work he has mapped out?—I am, &c.,

H. DROOP RICHMOND.

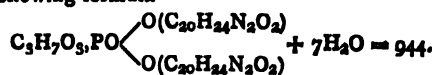
Rodbourne, Claremont Road,
St. Margaret's, June 18, 1898.

CHEMICAL NOTICES FROM FOREIGN SOURCES.

NOTE.—All degrees of temperature are Centigrade unless otherwise expressed.

Bulletin de la Société de Pharmacie de Bordeaux,
January, 1898.

Glycerophosphates of Quinine.—E. Falières.—Glycerophosphate of quinine has been assigned the following formula, $C_3H_7O_3PO_3(C_{20}H_{24}N_2O_2)_2$, and described as small colourless crystals, in the form of needles, easily and completely soluble in warm water and in alcohol; it contains 68 per cent of quinine. It should be noticed that this formula applies to an anhydrous salt. The author's experiments show that the crystallised salt contains from 7 to 10 molecules of water, according to the degree of saturation of the acid by one or two molecules of quinine. Theory foresees and experiment confirms the existence of two glycerophosphates of quinine, corresponding to the sulphates or hydrochlorates known (the basic and the neutral salt). The basic glycerophosphate of quinine has the following formula—



Practically, the preparation of the glycerophosphate with two molecules of quinine cannot be carried out by the double decomposition of an alkaline glycerophosphate and a salt of quinine. Like all the glycerophosphates, glycerophosphate of quinine must be obtained by precipitation in a dehydrating agent in which it is insoluble, such as ether. By preparing it under these conditions glycerophosphates of quinine takes the form of a very white, light, crystalline powder, unchangeable when exposed to the air, easily soluble in water acidulated with glycerophosphoric acid, soluble in warm water, in strong alcohol in the cold or in dilute warm alcohol, and insoluble in ether. It contains 1 molecule of acid, 2 molecules of quinine, and 7 molecules of water. The neutral glycerophosphate of quinine—



is a pale yellow crystalline powder, moist to the touch, having a horny appearance when deprived of part of its water of crystallisation. It melts at 120° into a vitreous

mass of a brownish colour. It is easily prepared by the double decomposition of an alkaline glycerophosphate and a neutral salt of quinine (sulphate or hydrochlorate); but the prolonged washings necessary to rid it of the sulphates or chlorides partly decompose it. Both these glycerophosphates may be estimated by dissolving in a mixture of hot alcohol and water, adding a few drops of phthalein and then a solution of decinormal potash, until the rose-colour remains permanent.

February, 1898.

A New, Very Sensitive, and Specific Reaction of Citric Acid. Detection in Vegetable Juices, Wine, Milk, &c.—G. Denigès.—In a previous communication the author briefly mentioned that the products of oxidation of citric acid, by manganic oxidising agents in an acid medium, gave an insoluble mercurial compound in the presence of mercuric sulphate. On further studying this reaction he finds that it is of extreme sensitiveness, and under certain conditions a specific test for citric acid. The method has been applied to numerous cases with excellent results.

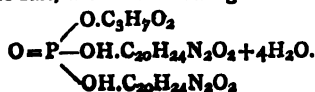
Estimation of Commercial Sulphate of Quinine.—L. Barthe.—A claim for priority by the author over a method described by M. Kubli in the *Mercur Scientificus*, February 1, 1897.

Journal de Pharmacie et de Chimie,
Series 6, vol. vii., No. 8.

The Physiology of Gentianose; its Splitting-up by Soluble Ferments.—E. Bourquelot.—It is well known that the cane-sugar formed in beetroot is, in the second vegetative period, which corresponds to the formation of grains, split up into assimilable sugars (dextrose and levulose) by a soluble ferment. An analogous splitting-up of gentianose in gentian has been shown to take place. Invertine only partially hydrolyses gentianose, while the liquid from *Aspergillus* does it completely.

Criticisms on the Volumetric Methods for the Estimation of the Phosphoglycerates.—H. Imbert and J. Pages.—After reviewing the work of many writers on this subject, the authors conclude:—1st, that the method of estimating the glycerophosphates proposed by MM. Imbert and Anstruc is sufficiently exact for all practical purposes, and can be generally used: 2nd, that—thanks to the use of chloride of calcium—the phosphates can be estimated in the presence of glycerophosphates, especially if the quantity reaches at least 5 per cent of the mixture; below this limit the operation is rather more delicate: 3rd, the boreates and silicates are not detected, and must be specially tested for, qualitatively.

Glycerophosphate of Quinine.—M. Moncour.—Glycerophosphate of quinine can be prepared by neutralising a titrated solution of glycerophosphoric acid by an equivalent quantity of quinine; or by the double decomposition of two solutions, one containing a salt of quinine, and the other glycerophosphate of lime; these solutions are used in equivalent proportions. The compound obtained is the same by both methods, and appears in the form of fine white needles closely resembling sulphate of quinine in appearance. It is inodorous and bitter, but not nearly so bitter as the sulphate. It is hardly soluble in water or cold alcohol, but readily so in hot alcohol and glycerin. The insolubility of glycerophosphate of quinine in water is not a very serious drawback, as the addition of a small quantity of citric or hydrochloric acid entirely overcomes it. This new salt of quinine is not altered in its composition by the dilution of its solutions, nor by heat, but, different from other glycerophosphates, is a very stable body; it melts at 154°. It is a basic salt, with the following definitive formula:—



Corresponding to the following composition:—

Quinine	72.64 per cent.
Glycerophosphoric acid ..	19.28 „
Water	0.09 „

Its richness in quinine is very near that of the other salts employed therapeutically; it is higher than the neutral salt, which only titrates 59.1 per cent, and is almost equal to the officinal sulphate which titrates 74.31 per cent; it can therefore be prescribed in the same doses, and in the same manner, while the presence of glycerophosphoric acid gives it the extra advantage of providing phosphorus in an easily assimilable condition, capable of accelerating nutrition and combating nervous depression.

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INDEX.

- ABATI, G.**, refraction and dispersive power of silicon in its compounds, 271
- Acetate of phenyl**, action of chloride of acetyl on, 236
- Acetic acid**, combinations of pyridine and of trimethylamine with, 249
- Acetone**, manufacture of oil of, 83
- Acetones and aldehyds**, reaction of, 236
- Acetylene**, sodium on, 177
- Acetylferufurane**, 45
- Acetylurethane**, ammonia and substituted ammonias on, 162
- Acid**, formaldehydtrioxyfluorondicarboxylic, 223
- lauronic**, 201
- pervanadic**, 201
- phosphoglycerates**, 285
- thorium oxalates**, composition of, 141
- Acids**, bromsuccinic, 116
- causes of reciprocal displacement of two, 188
- oxidation of certain, 234
- relation of taste of to degree of dissociation, 91
- Addison, W. L. T.**, atom forms, as deduced from the crystalline modifications of the elements, 251
- Adenin**, synthesis of, 165
- Adrian and Trillat, M.M.**, acid phosphoglycerates, 285
- estimation of phosphoglycerates, 141, 237
- phosphoglycerate of lime, 12
- reaction of phosphoric acid on glycerin, 237
- "Africa, Proceedings of the Chemical and Metallurgical Society of South,"** vol. I. (review), 248
- Africa**, sandstone off West Coast of, 119
- "Agriculture in some of its Relations with Chemistry"** (review), 163
- Air**, carbonic acid in, 165, 188
- chloride of palladium for detection of carbonic oxide in, 212
- composition of, 95
- new constituent of atmospheric, 287
- of towns, analysis of, 200
- refractivity of, 1
- spectral research on atmospheric, 288
- Alchemy**, revival of in France, 69, 73
- Alcohol**, dissociation of substances dissolved in aqueous, 189
- preparation of absolute, 52
- Aldehyde**, coloured reaction of ordinary, 29
- Aldehyde of ammonia**, 71
- Aldehyds and acetones**, reaction of, 236
- and nitrogen, 164
- "Alembic Club Reprints, No. 13, The Early History of Chlorine"** (review), 70
- "Alembic Club Reprints, No. 14, Researches on Molecular Asymmetry"** (review), 79
- Alfa, J., and R. F. Weinland**, fluosilicate and fluophosphate of potassium and rubidium, 165
- Alkaline carbides**, formation of, 85
- sulphantimonites, 274
- Alkalies**, action of on amides, 57
- Alkyl bismuth iodides** and bismuth iodides of vegetable bases, 138
- iodides on silver malate and on silver lactate, 163
- Allyldipropylcarbinol**, trivalent alcohol of, 129
- Allylethylphenylcarbinol**, 129
- Allylmethyltertiarybutylcarbinol**, 189
- Aluminium** as a reducing agent, 213
- chloride on camphoric anhydride, 210
- corrosion of, 207
- and its alloys, impurities in, 45
- in qualitative analysis, 105
- mercury couple, 58
- separation of, 179, 265
- American and Indian podophyllum**, 113
- Association for the Advancement of Science, 214
- Amide and thioamide compounds**, oxidation of, 201
- Amides**, action of alkalis on, 57
- Amido-compounds**, di-isocyanates upon, 162
- Amido-oxytutidines**, production of, 124, 125
- Amines of the naphthalene series**, action of formaldehyds on, 258
- Ammonia and ammonium nitrate**, equilibrium between, 189
- apparatus for determining composition of, 203
- drying of, 198
- in a gaseous atmosphere, detecting, 267
- rate of escape of from aqueous solution, 79
- test of Coccaium hydrochloricum, 183
- Ammoniacal bromides of silver**, 261
- compounds, oxidation of, 71
- Ammonium aldehyde**, 23
- cyanate, solid, 209
- nitrate and ammonia, equilibrium between, 189
- Ammonium peroxide**, 165
- salts of tartaric and citric acids, lead-free, 119
- Amorphous mercurous iodide**, colour of, 40
- "Analysts' Laboratory Companion"** (review), 104
- Analysis of bearing-metal alloys**, 45, 52
- Analytical work**, dignity of, 173, 185, 193
- André, G.**, behaviour of pyridine with propionic, acetic, and formic acids during distillation, 45
- combinations of pyridine and of trimethylamine with formic and acetic acids, 249
- galacyl, 286
- Andreasch, R.**, thiourea derivatives, 165
- Andrews, W. W.**, plaster of Paris method in blowpipe analysis, 15
- Anhydride**, formation of, 116
- Anhydrous fluoride of glucinum**, 288
- hydrogen cyanide, preparation of, 124
- sulphide of barium, 140
- Anhydroxycobaltamines and oxycobaltamines**, 201
- Anilidoximes**, action of hydroxylamines on, 225
- Austruc, A., and H. Imbert**, neutralisation of glycerophosphoric acid by the alkalis, 36
- Antimony**, volumetric estimation of, 36
- Antipyrin**, compounds of with the aldehyds, 47
- combination of with the aldehyds, 95
- iodine on, 141
- Aqueous solution**, rate of escape of ammonia from, 79
- Arctostaphylos uva ural**, yellow colouring matter of leaves of, 208
- Argon**, refractivity of, 1
- Armitage, F. P.**, atomic weight of boron, 78
- Arnaud, M.**, products from splitting-up ouabaine by hydrolysis, 285
- researches on ouabaine, 83
- Aromatic diurethanes and piperazine**, 45
- nitroso-bases**, diazomethane on, 226
- urethanes of conicine, 116
- "Arrangement of Atoms in Space"** (review), 273
- Arsenic**, lead, and copper, separation of, 172
- Asbestos** combustion furnaces, 51
- Aspillerus "niger"**, nitrate, sulphate, chlorate, and phosphate of ammonia on, 45
- Assay of minium**, 250
- Aston, Miss E., and P. A. Guye**, temperature on rotatory power of liquids, 10
- Astruc, A.**, glycerophosphates, 72
- Atlantic Ocean**, composition of, 155, 171
- Atmosphere**, new constituents of, 295
- Atom forms**, 251
- "Atoms in Space, The Arrangement of"** (review), 273
- Atomic weight of boron**, 104
- cobalt, 20, 30
- praseodymium and of neodymium, 280, 292
- zirconium, 221, 231
- weights of hydrogen, nitrogen, and carbon, values of, 224
- report of committee on, 239
- Auric chloride**, decomposition of, 74
- Austin, M.**, estimation of manganese, 242
- and F. A. Gooch, estimation of manganese as the sulphate and as the oxide, 255
- condition of oxidation of manganese precipitated by the chloride process, 260, 278
- Australasian Association for the Advancement of Science**, 12
- Ayrton, W. E., and T. Mather**, galvanometers, 246
- Asimido-compounds of benzimidazole**, 226
- BACILLUS tartaricus**, 106
- Bailey, G. H.**, "The Tutorial Chemistry, Part II., Metals" (review), 200
- Baker, H. B.**, drying of ammonia and hydrogen chloride, 198
- Bancroft, W. D.**, "The Phase Rule" (review), 34
- Barley straw**, carbohydrates of, 197
- testing for diastase from, 12
- Barral, E.**, chloridised derivatives of carbonate of phenyl, 201
- Barthe, L.**, commercial sulphate of quinine, 298
- Barton, E. H.**, attenuation of electric waves along a line of negligible leakage, 283
- Basic ortho-antimonates of potassium**, 71
- Basilio**, essence of, 237
- Baskerville, O., and F. W. Miller**, decomposition of sulphuric acid, 191
- reactions between mercury and concentrated sulphuric acid, 49

- Bater, C. E., "Brewing Calculations: Gauging and Tables" (review), 23
- Battandier, J., and T. Malosse, retamine, 11
- Battery with carbon electrodes, 83
- Baugé, G., double carbonate of soda and protoxide of chromium, 45, 177
- Baxter, G. F., and T. W. Richards, revision of the atomic weight of cobalt, 20, 30
- Bayerlein, H., and H. Fresenius, estimation of chromium in chrome-iron ore, 141
- Bayley, T., cyclical law of the elements, 157
- Bearing-metal alloys, analysis of, 41, 52
- Beatty, W. A., and J. H. Kastle, effect of light on combination of hydrogen and bromine, 111
- Beckh, W., syntheses with chlorfumaric ester, 152
- Beech tar, constituents of, 152
- Behal, A., new cyclic ketones, 35, 59
- Belugou, G., heat of neutralisation of ethyl-phosphoric acid, 262
- and H. Imbert, heat of neutralisation of glycerophosphoric acid, 36
- Bentley, W. H., and R. Hertz, oxidation of paranitrotoluene-sulphonic acid to dinitroethylbenedisulphonic acid and to paranitrobenzaldehydortho-sulphonic acid, 236
- Benzene, absorption bands in spectrum of, 103
- action of bromine on, 125
- combination of phosphoric anhydride with, 123
- hexabromide, 125
- vapour and ethylene, separation of, 105
- Benzimidazole, azimido compounds of, 226
- Benzophenone, derivatives of, 198
- sulphonation of, 210
- Benzylhydrazide, action of on glucose, 152
- Bernfeld, I., metallic sulphides as electrodes, 189
- Berthelot, D., absorption of oxygen by pyrogallate of potash, 249
- action of oxygen on sulphide of carbon, and chemical influence of light, 248
- chemical actions of electric discharge, 140, 142, 164, 165, 188
- melting points of silver and gold, 115
- values of atomic weights of hydrogen, nitrogen, and carbon, 224
- Bessler, H., and A. Claus, naphthoquinolines, 188
- Besson, A., phosphorus oxide, 20
- Bevan, E. J., C. Smith, and C. F. Cross, action of hydrogen peroxide on carbohydrates, 238
- carbohydrates of barley straw, 197
- "Bibliography of the Metals of the Platinum Group" (review), 95
- Bicarbonates, carbonic acid in, 109
- Bismuth iodides of vegetable bases, 138
- salts and mercury, separation of, 165
- transparency of in a magnetic field, 116
- Blaise, E. E., preparation and etherification of dissymmetrical dimethylsuccinic acid, 157
- synthesis of terebic acid, 83, 286
- Bleier, O., gasometric apparatus, 225
- Blowpipe analysis, plaster of Paris method in, 15
- Bodroux, F., ether oxides of naphthol, 188
- Boehringer, C. F., and M. Soehne, ammonia test of Calcium hydrochloride, 183
- Bogorodsky, A., trivalent alcohol of allyldipropylcarbinol, 129
- and J. Ljubarsky, allylethylphenylcarbinol, 129
- Boiling point apparatus, 59
- Bolton, H. C., hysterical chemistry, 3, 16
- revival of alchemy in France, 69, 73
- Borates, action of carbon dioxide on soluble, 294
- Borocarbide of glaucinum, 289
- Boron, atomic weight of, 78, 104
- Bošnjakovic, D. S., evaporating funnel, 141
- Boudouard, O., cerium, 27
- neodymium, 193
- and P. Schutzenberger, yttric earths in monazite sands, 193, 204, 220, 229
- Bougault, J., action of iodine on antipyrine, 141
- Bourquelot, E., physiology of gentianose, 298
- and L. Nardin, preparation of gentianose, 71, 271
- Bouveault, L., acetylfurfuran and its presence in Stockholm tar, 45
- constitution of camphoric acid, 83
- derivatives of guaiacol, 46
- phenol-glyoxylic acids, 46
- Brame, J. S. S., and J. W. Rodger, optical rotations of methyl and ethyl tartrates, 163
- Brauner, B., atomic weight of thorium, 160
- chemistry of thorium, 160
- compound nature of cerium, 160
- praseodymium and neodidymium, 161
- Brearley, H., separations from chromic acid, 49, 131, 179, 216
- "Brewing Calculations: Gauging and Tables" (review), 23
- Briggs, W., "General Elementary Science" (review), 223
- "The Tutorial Chemistry—Part II. Metals" (review), 200
- Brissemoret, M., solubility of theobromine in aqueous solutions of salts, 153
- British Association, 271
- Fire Prevention Committee, 106
- Brittain, C. E., and J. B. Cohen, action of alkalis on amides, 57
- Bromacetal on the sodium derivative of ethylic malonate, 210
- Bromanile, action of cyanamide on, 116
- Bromic acid, reduction of, 149
- Bromine, action of on benzene, 125
- and hydrogen, effect of light on combination of, 111
- chlorine, and iodine, separation and estimation of, 67
- derivatives of naphthylamines, 128
- separation and estimation of, 276
- Brominated ketones, 166
- Bromosuccinic acid, 116
- Bromotolylhydrazine, derivatives of, 36
- Brooks, F. C. H., a curious lead salt, 191
- Browning, K. C., and S. Ruhemann, formation of ethylic dihydroxydinicotinate from ethylic cyanacetate, 115
- Bryan, G. H., electro-magnetic induction, 81
- Bryant, E. G., "A Graduated Course of Chemical Problems" (review), 104
- interaction of magnesium and solution of sulphate of copper, 139
- Buisine, A., and P., manufacture of oil of acetone, 83
- Buisson, H., transparency of bismuth in a magnetic field, 116
- Bunte, H., lighting effect of flames, 131
- Butler, C. F., and E. Edser, method of reducing prismatic spectra, 260
- CADMIUM**, determination of, 93
- in vacuo, spectrum of, 71
- "Calorific Power of Fuels," Founded on Scheurer-Kestner's "Pouvoir Calorifique des Combustibles" (review), 176
- Calvert, H. T., and J. B. Cohen, aluminium mercury couple, 58
- formation of monomethyl-aniline, 57
- Camphor and its derivatives, 70, 82
- stereoisomeric derivatives of, 259
- sulphonic derivatives of, 177
- Camphoric acid, constitution of, 83
- anhydride, aluminium chloride on, 210
- Cannabinol, 150
- Carbide of calcium, use of for preparation of absolute alcohol, 52
- of magnesium, formation of, 85
- of sodium, 35
- and monoacetic acetylene, preparation of, 177
- Carbides, method of preparing, 11
- of barium and manganese, dissociation of, 165
- Carbohydrates, action of hydrogen bromide in presence of ether on, 282
- hydrogen peroxide on, 233
- nitrification of, 152
- of barley straw, 197
- Carbon compounds, ultra-violet absorption spectra of, 103
- dioxide, action of on soluble borates, 294
- and hydrogen, determination of, 215
- estimation of, 140
- hydrogen, and nitrogen, values of atomic weights of, 224
- in iron, 105
- monoxide on platinum and palladium, 105
- preparation of, 124
- Carbonate of menthol, preparation of, 242
- of phenyl, chloridised derivatives of, 201
- of soda, and protoxide of chromium, 177
- Carbonic acid, conversion of carbonic oxide into, 212
- in air, 165, 188
- bicarbonates, 109
- ethers from the phenols, piperidine on, 36
- oxide, conversion into carbonic acid, 212
- in air, chloride of palladium for detection of, 212
- on palladium chloride, 140
- reaction of reagents on, 200
- Carnot, A., separation and estimation of iodine, chlorine, and bromine, 67, 276
- Casein in milks, estimation of, 108
- Cause, H., action of ethylic aldehyde on phenylhydrazine, 236
- Cause, H., estimation of phenylhydrazine, 232
- volumetric estimation of anti-mony, 36
- Caustic soda, analysis of, 229
- Cavalier, J., phosphoric di-ethers, 285
- phosphoric mono-ethers, 274
- Cazenave, P., and M. Moreau, aromatic diurethanes and piperazines, 45
- aromatic urethanes of coni-cine, 116
- piperidine on carbonic ethers from the phenols, 36
- Schiff reaction applied to some substituted fuchsin, 83
- Cerite, earths of, 97
- metals, new compounds of, 75
- Cerium, 27
- and thorium, separation of oxides of, 245, 254
- atomic weight of, 45
- compound nature of, 160
- Chassevant, A., new water ureometer, 105
- Chemical action brought about by electric discharge, 140
- exercised by electric discharge on organic compounds, 140
- of the electric discharge on dielectric liquids, 165
- actions of electric discharge, 140, 142, 164, 188
- "Chemical and Metallurgical Society of South Africa, Proceedings" (review), 248
- "Chemical Problems, a Graduated Course of" (review), 104
- Chemical preparations, analysis of, 121
- reactions, irreversibility in, 23
- temperature on, 274
- Society, 7, 54, 78, 100, 113, 124, 148, 160, 175, 197, 207, 233, 257, 282
- dinner, 260
- Chemically inactive elements, 87
- "Chemistry, First Year's Course of Experimental Work in" (review), 224
- "Chemistry for Photographers" (review), 44
- "Chemistry, the Tutorial, Part II., Metals" (review), 200
- Chemistry, hysterical, 3, 16
- Institute of, 46
- Chloral hydrate, condensation of with orcinol, 207
- Chloride of acetyl, action of on acetate of phenyl, 236
- of benzoyl- β -nitrated on mono-substituted ortho-diamines, 96
- of palladium for detection of carbonic oxide in air, 212
- Chlorine, action of on pyridine, 236
- derivatives of naphthylamines, 128
- of pyridine, 210
- iodine, and bromine, separation and estimation of, 67
- separation and estimation of, 276
- Chlorobrombenzene, 103
- Chlorfumaric ester, syntheses with, 152
- "Chlorine The Early History of" (review), 70
- Chlorocyanamide, 10
- Chloroform, decomposition of, 165
- and alkaline hydroxides on nitrobenzoic acid, 58
- Chlorhydrate of cocaine, rotatory power of, 106
- Chloropyridine carboxylic acids, production of, 283
- Choline and trigonelline in seeds of strophanthus, and preparation of strophanthine, 225
- Chrome-iron ore, estimation of chromium in, 141
- Chromic acid, separations from, 49, 131, 179, 216
- Chromium in chrome-iron ore, estimation of, 141

- Chromium, determination of in iron and steel, 187
estimation of, 156
silicic of, 188
Chromosulphochromic acids, 45
Cinchonine, derivatives of, 140
Cis- and trans-carbonic acid, synthesis of, 209
Citric acid, reaction of, 298
City and Guilds of London Institute, 24, 72
Clark, E., and G. Young, ammonia and substituted ammonia on acetylurethane, 162
Clarke, F. W., report of committee on atomic weights, 239
Classen, A., and W. Löb, "Quantitative Chemical Analysis by Elektrolysis" (review), 176
Claus, A., and H. Beeseler, naphthoquinolines, 188
and O. Jack, chlorine and bromine derivatives of naphthylamines, 128
Clowes, F., and J. B. Coleman, "Quantitative Chemical Analysis" (review), 104
"Coal-gas, Commercial Uses of" (review), 44
Cobalt, atomic weight of, 20, 30
constitution of, 213
Cocaine hydrochloride, ammonia salt of, 183
Cockburn, G. E., and J. A. Gardner, researches on the terpenes, 37
Cohen, E., dissociation of substances dissolved in aqueous alcohol, 189
new method of element change, 274
J. E., and C. E. Brittain, action of alkalis on amides, 37
and H. T. Calvert, aluminium-mercury couple, 58
formation of monomethylaniline, 37
Coleman, J. B., and F. Clowes, "Quantitative Chemical Analysis" (review), 104
Collie, J. N., and O. C. Frye, action of bromine on benzene, 123
and Miss L. Hall, production of some nitro- and amido-oxylutidines, 123
and T. Tickle, production of some nitro- and amido-oxylutidines, 124
and W. Lean, production of some chloropyridinecarboxylic acids, 283
Collinson, R. W., and W. H., Perkin, lauronic acid, 210
Colouring-matters of Indian dyestuffs, Delphinium zaili, 126
Colson, A., causes of reciprocal displacement of two acids, 188
irreversibility in chemical reactions, 23
temperature in chemical reactions, 274
"Commercial Fertilisers and Chemicals" (review), 211
Commercial incandescent bodies, investigation of, 249
Conicine, aromatic urethanes of, 116
Conjectural group of inactive elements, 120
Cook, E. H., "First Year's Course of Experimental Work in Chemistry" (review), 224
Copper, arsenic and lead, separation of, 172
compounds of dicarboxylglutamic ester, 165
impurities in raw, 72
method for determining, 41, 53
sulphate, interaction of magnesium and solution of, 127
Coppock, J. B., Gladding's method for phosphoric acid, 242
Corundum deposits in Eastern Ontario, 142
Cotton-seed oil, reaction of, 11
Cousin, H., new derivatives of homopyrocatechin, 249
pyrocatechin, 106
Crofts, J. M., molecular weights of permanganates, perchlorates, and periodates in solution, 236
and R. S. Morrell, emolic and ketonic forms of ethylic aceto-acetate, 235
ferric chloride on the ethereal salts of ketonic acids, 149
Crookes, Sir W., and J. Dewar, London water supply, 33, 90, 122, 185, 232, 278
Crookes tube, circulation of gaseous matter in, 150
tubes, circulation of residual gaseous matter in, 260
novel facts in, 23
Crosa, C. F., E. J. Bevan, and C. Smith, action of hydrogen peroxide on carbohydrates, 233
carbohydrates of barley straw, 127
Cryoscopy and osmotic pressure, 174
Crystalline liquids, investigation of, 286
Crystallised silicon, electrical resistance of, 71
carbon dioxide, 216
Cundall, J. T., lecture experiment, 102
Cupellation versus scorification, 39
Cuprous sulphate, existence of, 23
Curie, Mme. S., rays emitted by compounds of uranium and of thorium, 249
Cyanamide, action of on bromanile, 116
Cyanides, interaction of with thiosulphates, 131
Cyanogen, addition of to sodium salt of malonic acid, 166
Cyclic ketone, a new, 39
Cyclical law of the elements, 157
DE CONINCK, O., action of oxidants on nitrified bodies, 225
decomposition of sulphocyanic ethers, 188
oxidation of amide and thioamide compounds, 201
oxyptomaine, 140
De Forcrand, M., aldehyde of ammonia, 71
De Gramont, A., dissociation spectra of melted salts, 28, 88
spectral analysis of non-conducting compounds, 218
De Koningh, L., estimation of rosin and rosin oil in linseed oil, 287
mineral matter in rubber goods, 74
preparation of lead-free ammonium salts of tartaric and citric acids, 119
De la Source, L. M., filter-paper, 12
Decomposition of some substances under influence of electric vibration, 274
Deerr, N., molecules and liquefaction heats, 35
Defacqz, E., iodide of tungsten, 204
impurities in aluminium and its alloys, 45
Delacroix, A., basic ortho-antimonates of potassium, 71
Delépine, M., ammonium aldehyde, 23
hydramides and isomeric bases, 85
hydrocinnamide, 139
isocoumarin and tetrahydroisocoumarin, 224
oxygen on dissolved formic aldehyd, 45
quinoleic bases, 212
Delphinium zaili, colouring-matters of, 126
Demarcay, E., spectrum and nature of neodmium, 219
Demoussy, E., oxidation of ammoniacal compounds, 71
Denigès, G., combination obtained with nitrate of mercury and trimethylcarbinol, 225
detecting ammonia in a gaseous atmosphere, 267
estimation of casein in milks, 106
general reaction of the ethenic carbides, 261
reaction of citric acid, 298
Dennstedt, M., and W. Göblich, preparing hydronitric acid, 181
Desgrez, A., and M. Nieloux, decomposition of chloroform, 165
Deslandres and Moissan, M.M., spectral research on atmospheric air, 288
Dewar, J., boiling-point and density of liquid hydrogen, 261, 282
liquefaction of hydrogen and helium, 216, 257
and J. A. Fleming, determinations of dielectric constants of organic bodies and electrolytes, 13, 25, 37
and Sir W. Crookes, London water supply, 33, 90, 122, 185, 232, 278
Diastase, chemical nature of, 225
from barley, testing for, 12
Diastatic fungi, 137
Diastatic substances, testing, 173
Diazo-compounds of phenylenediamine, 166
Diazomethane on aromatic nitroso-bases, 226
on nitrosophenol, 226
Dibasic organic acids, dissociation of, 274
Dicamphene, crystallised hydride of, 116
Dicarboxylglutamic ester, copper compounds of, 165
Dichloroacetyl groups, effect of on rotatory power of methylic and ethylic glycerates and tartrates, 80
Dielectric constants of organic bodies and electrolytes, determination of, 13, 25, 37
liquids, chemical action of electric discharge on, 165
Diethylic monobenzoyl and monotoyl tartrates, comparative rotatory powers of, 162
Dihydroxyfumaric acid, affinity constants of, 235
Dihydroxymaleic acid, affinity constants of, 235
Dihydroxypyridine, formation of, 162
Dihydroxytartaric acid, affinity constants of, 235
properties and relationships of, 234
Di-isocyanates upon amido-compounds, 162
Dilution, universal applicability of law of, 189
Dimethylaniline, end-product of action of nitrogen chloride on, 225
Dimethylsuccinic acid, preparation and etherification of, 137
Dinitrostilbenedisulphonic acid, oxidation of paranitrotoluenesulphonic acid to, 236
Diphenylmethane, sulphonation, 210
"Directory of Paper-makers," 166
Dissociation, degree of heat of solution and solubility, relationship between, 189
spectra of melted salts, 28, 88
Distilled waters, preparation of, 12
Divers, E., interaction of magnesium and solution of copper sulphate, 127
Dixon, F., and J. T. Hewitt, condensation of chloral hydrate with orcinol, 207
Dobbie, J. J., and A. Gray, connection between electrical properties and chemical composition of different kinds of glass, 143
and A. Lauder, Kopfer's method for determination of carbon and hydrogen, 215
and F. Marsden, o-chlorobromobenzene, 103
and W. N. Hartley, absorption bands in spectrum of benzene, 103
ultra-violet absorption-spectra of some closed chain carbon compounds, 103
Dobbin, L., interaction of cyanides with thiosulphates, 131
sandstorm off West Coast of Africa, 119
Dobriner, E., and W. Schranz, analysis of caustic soda, 229
sulphide and sulphhydrate of sodium, 108
Dogfish, liquid fatty acids of fat of, 121
Dootson, F. W., and W. J. Sell, action of chlorine on pyridine, 236
chlorine derivatives pyridine, 210
Dowdard, E., estimation of starch in flour, 107
Drechsel, E., a natural silicic ether, 214
Drouin and Potain, M.M., chloride of palladium for detection of carbonic oxide in the air, and conversion of this gas into carbonic acid, 212
Duclaux, M., contamination of wells, 23
Dudley, C. B., dignity of analytical work, 173, 185, 195
Dunstan, W. R., and T. A. Henry, Indian and American podophyllum, 113
orycannabin from Indian hemp, 114
wood of Goupia tomentosa, 114
Dupont, J., and M. Guerlain, essence of basilic, 237
Dutremblay and Lugan, M.M., preparation of oxygen, 11
Dynamical illustrations of certain optical phenomena, 128
EASTERFIELD, T. H., T. B. Wood, and W. T. N. Spivey, cannabinol, 150
Edser, E., and C. P. Butler, method of reducing prismatic spectra, 260
Eder, J. M., and E. Valenta, line spectrum of silicon, 206
Edinburgh University Chemical Society, 82
Edmed, F. G., constitution of oleic acid, 259
Eiermann, K., diazo-compounds of phenylenediamine, 166
Eiloart, A., "The Arrangement of Atoms in Space" (review), 273
Elaidic acid, action of sulphuric acid on, 129
Elbe, M., persulphuric acid, 99
Electric currents, passage of in mixed solutions of electrolytes, 190
discharge, chemical action brought about by, 140, 142, 164, 165, 188
signalling without connecting wires, 58
vibration, decomposition of some substances under influence of, 274
waves, attenuation of along a line of negligible leakage, 283

- Electricity, influence of discharge of on atmospheric air, 102
 "Electro-chemistry, the Elements of Treated Experimentally" (review), 248
 Electro-magnetic induction, 81
 Electrodes, metallic sulphides as, 189
 working of dropping, 270
 Electrolytes, determinations of dielectric constants of, 13, 25, 37
 dissociation of, 243
 passage of electric currents in mixed solutions of, 190
 Element change, new method of, 274
 Elements, atom forms, as deduced from the crystalline modifications of the, 251
 conjectural group of inactive, 120
 cyclical law of, 157
 "Elements of Electro-chemistry Treated Experimentally" (review), 248
 "Elementary Course of Practical Organic Chemistry" (review), 224
 Elliott, W. J., chloroform and alkaline hydroxides on nitrobenzoic acids, 58
 Emerald, treatment of by the electric furnace, 285
 Enamels of high dilatation, 272
 Erdmann, M., preparation of carbonate of menthol, 242
 Essence of basilic, 237
 Esters, formation and hydrolysis of, 7
 Etard, A., and G. Meier, crystallised hydride of dicamphene, 116
 Ethenic carbides, general reaction of, 261
 Ether oxides of naphthol, 188
 Ethylene and benzene vapour, separation of, 105
 Ethylic acetate, enolic and ketonic forms of, 235
 aldehyd, action of on phenylhydrazin, 236
 dihydroxydinicotinate, formation of, 115
 di-monochloracetyl tartrates, rotation of, 80
 glycerates, effect of mono-, di-, and tri-chloracetyl groups on rotatory power of, 80
 malonate, bromacetal on sodium derivative of, 210
 Ethyl-phosphoric acid, heat of neutralisation of, 262
 Ethylpiperidine and its methyl derivative, 225
 Ethyl tartrates, optical rotations of, 163
 Evans, W. T., and W. A. Shennstone, influence of discharge of electricity on atmospheric air, 102
 Everett, J. D., dynamical illustrations of certain optical phenomena, 128
- FABRY, C., and A. Perot, interferential spectroscopy, 82
 Falières, E., glycerophosphates of quinine, 297
 Fats, detection of yellow azo-dye for colouring, 112
 Fehling's solution, 57
 Feilmann, M. E., and J. J. Sudborough, formation and hydrolysis of esters, 7
 Fenton, H. J. H., oxidation of certain acids, 234
 properties and relationships of dihydroxytartaric acid, 234
 volumetric estimation of sodium, 78
 and Mildred Goettling, action of hydrogen bromide in presence of ether on carbohydrates, 288
 Fergusson, 64
- Ferric chloride, chloridising action of in the aromatic series, 285
 on the ethereal salts of ketone acids, 149
 Ferrosodium, 192
 Ferrous salts, absorption of nitric oxide by, 268, 290
 Ficquet, L., and L. Grimbart, new ferment from tartrates, 106
 Filter-paper, 12
 Fink, E., carbonic oxide on palladium chloride, 140
 Fire Prevention Committee, British, 106
 Fischer, R., synthesis of adenin, 165
 Flames, lighting effect of, 151
 Fleming, J. A., and J. Dewar, determination of dielectric constants of organic bodies and electrolytes, 13, 25, 37
 Fletcher, T., "Commercial Uses of Coal-gas" (review), 44
 Flour, estimation of starch in, 107
 Fluorescent substance, 226
 Fluorite, polymorphism of, 116
 Fluosilicate and fluophosphate of potassium and rubidium, 165
 Fock, A., dissociation in mixed salt solutions, 189
 Foods, salicylic acid in, 136
 Formaldehyde, condensation of, 114
 products of action of on gallic acid, 225
 testing of, 94, 98
 Formaldehyde, action of on amines of the naphthalene series, 258
 Formaldehydtrioxyfluorocarbonylic acid, 225
 Formic acid, combinations of pyridine and of trimethylamine with, 249
 aldehyd, oxygen on dissolved, 45
 Forster, M. O., camphor and its derivatives, 82
 isomeric bornylamines, 197
 Fortey, Emily C., hexamethylene and its derivatives, 207
 Franck, L., aluminium as a reducing agent, 213
 Francois, M., colour of amorphous mercurous iodide, 50
 Frankland, P., and J. McCrae, position-isomerism and optical activity, rotatory powers of diethylic mono-benzoyl and mono-toluy tartrates, 162
 and T. S. Patterson, effect of mono-, di-, and tri-chloracetyl groups on rotatory power of methylic and ethylic glycerates and tartrates, 80
 and A. Turnbull, rotation of ethylic and methylic di-monochloracetyl tartrates, 80
 "Freezing-point, Boiling-point, and Conductivity Methods" (review), 212
 Freezing-point in very dilute solution, method of determining, 8
 "French Self-taught, with Phonetic Pronunciation" (review), 296
 Fresenius, H., and H. Bayerlein, estimation of chromium in chrome-iron ore, 141
 Fritzsche, Dr. P., determination of the weight of smoke, 249
 Frye, C. C., and J. N. Collie, action of bromine on benzene, 125
 Fuchsine, Schiff reaction applied to some substituted, 82
 "Fuels, the Caloric Power of" (review), 176
 Fungi, diastatic, 137
 Funnell, evaporating, 141
 Furfuralcroleins and furfurols, condensation products of, 225
 Furfurols and furfuralcroleins, condensation products of, 225
- GAÏACYL, 286
- Galbraith, W., determination of chromium in iron and steel, 187
 Gallic acid, products of action of formaldehyd on, 225
 Galvanometers, 246
 Gardner, J. A., and G. B. Cockburn, researches on the terpenes, 57
 Garrett, F. C., and A. Harden, "Elementary Course of Practical Organic Chemistry" (review), 224
 Garrigue, W. E., analysis of bearing-metal alloys, 41, 52
 Gas and gases, 284
 the new, 297
 "Gas Engineers' Pocket-book" (review), 224
 Gases, density of, 95
 mixture of, 71
 and vapours, dissociation and polymerisation of, 23
 Gaseous atmosphere, detecting ammonia in, 267
 Gasometric apparatus, 225
 Gautier, A., carbonic acid in air, 188
 reaction of reagents on carbonic oxide, 200
 Geisler, J. F., test for detection of a yellow azo-dye for colouring fats, 112
 "General Elementary Science" (review), 223
 Genth, F. A., salicylic acid in foods, 136
 Gentianose, physiology of, 298
 preparation of, 71, 271
 George, G., apparatus for determining the composition of ammonia, sulphur dioxide, water, &c., 203
 Geyser action, new theory of, 103
 Gin and Leleux, M. M., dissociation of carbides of barium and manganese, 165
 Giran, H., combination of phosphoric anhydride with benzene, 123
 Gladding, T. S., phosphoric acid determination, 32
 Gladding's method for phosphoric acid, 242
 Glaser, C., composition of acid thorium oxalates, 141
 sodium peroxide in quantitative analysis, 123
 Glass, connection between electrical properties and chemical composition of different kinds of, 143
 Glucinum, anhydrous fluoride and oxyfluoride of, 288
 borocarbide of, 289
 iodide of, 266
 preparation of, 172
 alloys of, 44
 Glucose, action of benzhydrazide on, 152
 sugar, manufacture of, 106
 Glycerin, reaction of phosphoric acid on, 237
 Glycerophosphates, 72
 of quinine, 297, 298
 Glycerophosphoric acid, heat of, 26
 neutralisation by the alkalis, 95
 Glyoxalidines, 83
 Gnadin, A., allylmethyltertiarybutylcarbinol, 189
 Göblich, W., and M. Dennstedt, preparing hydronitric acid, 181
 Gold and silver, melting-points of, 115
 deposited on iron, 129
 Goldschmidt, H., relationship between heat of solution, solubility, and degree of dissociation, 189
 Gomberg, M., isonitramin and nitrosobutyric acid, 117
- Gooch, F. A., and Martha Austin, condition of oxidation of manganese precipitated by the chlorate process, 269, 276
 estimation of manganese as the sulphate and as the oxide, 255
 Goettling, Mildred, and H. J. H. Fenton, action of hydrogen bromide in presence of ether on carbohydrates, 282
 Gottig, C., products of explosive decomposition of nitro-compounds, 152
 Granger, A., metallic phosphides, 227
 Grape-sugar and milk-sugar estimating, 141
 Gray, A., and J. J. Dobbie, connection between electrical properties and chemical composition of different kinds of glass, 143
 Grimaux, E., derivatives of cinchonine, 140
 tetramethyl-diamido-benzophenone, 273
 Grimbart, L., and L. Ficquet, new ferment from tartrates, 106
 Grinberg, S., and F. Haber, electrolysis of hydrochloric acid, 213
 Guerlain, M., and J. Dupont, essence of basilic, 237
 Guaiacol, derivatives of, 46
 Gulchard, M., reduction of molybdic anhydride by hydrogen, 45
 Guilds and City of London Institute, 24, 72
 Guye, P. A., and Miss E. Aston, temperature on rotatory power of liquids, 10
 Guyot, A., and A. Haller, constitution of phthalic green, 44
 Gypsum, anhydrous sulphide of lime produced by dehydration of, 83
- HABER, F., and S. Grinberg, electrolysis of hydrochloric acid, 213
 Haga, T., and C. Harries, methylating of hydrazin hydrates, 152
 Hall, Miss L., and J. N. Collie, production of some nitro- and amido-oxylidines, 125
 Haller, A., and A. Guyot, constitution of phthalic green, 44
 Halphen, G., reaction of cotton-seed oil, 11
 Hampe, W., "Fafeln zur Qualitativen Chemischen Analyse" (review), 297
 Hamy, M., spectrum of cadmium in vacuo, 71
 Hantzsch, A., nitramine and isonitramine and their ethers, 165
 Harbeck, E., and G. Lange, carbon monoxide on platinum and palladium, 105
 estimating carbon in iron, 105
 separation of ethylene and benzene vapour, 105
 Harden, A., and F. C. Garrett, "Elementary Course of Practical Organic Chemistry" (review), 224
 Harries, C., constituents of beech tar, 152
 and T. Haga, methylating of hydrazin hydrates, 152
 Hartley, W. M., and J. J. Dobbie, absorption-bands in spectrum of benzene, 103
 ultra-violet absorption spectra of some closed chain carbon compounds, 103
 and H. Ramage, analysis of samples of metals, of chemical preparations, and minerals from Stassfurt potash beds, 121

- Hausser, J., sterilisation of liquids by filtration, 188
- Haworth, E. W., and W. H. Perkin, condensation of formaldehyd with ethylic malonate, 114
- Health Exposition, International, 83
- Helium, homogeneity of, 61
liquefaction of, 216, 237
refractivity of, 1
separation from a natural compound, 190
- Hempel, W., sodium, magnesium, and aluminium in qualitative analysis, 105
- Henry, T. A., and W. R. Dunstan, Indian and American podophyllum, 113
oxy-cannabim from Indian hemp, 114
wood of *Goupia tomentosa*, 114
- Hentschel, W., end-product of action of nitrogen chloride on dimethylaniline, 235
- Herissey, H., rotatory power of chlorhydrate of cocaine, 106
- Hertz, R., and W. H. Bentley, oxidation of paranitrotoluenesulphonic acid to dinitrotoluenedisulphonic acid and to paranitrobenzaldehydortho-sulphonic acid, 236
- Heuse, O., hydrocinchonine, 117
- Hewitt, J. T., and F. Dixon, condensation of chloral hydrate with orcinol, 207
and F. G. Pope, derivatives of bromotolylhydrazine, 56
- Hexamethylene and its derivatives, 207
- Hinsberg, O., and A. Simcoff, synthesis of naphthindol derivatives, 235
- Hintz, E., investigation of commercial incandescent bodies, 249
- Hixon, H. W., "Notes on Lead and Copper Smelting and Copper Converting" (review), 43
- Hofmann, K. A., and F. Kuspert, estimating hydroxylamine and hydrazine, 132
- Homopyrocatechin, new derivatives of, 249
- Hopfgartner, K., passage of electric currents in mixed solutions of electrolytes, 190
- Howe, J. L., "Bibliography of the Metals of the Platinum Group, 1748-1896" (review), 95
- Hydramides and isomeric bases, 83
- Hydrazinacetic acids, 166
- Hydrazine, estimating, 132
hydrates, methylating of, 132
- Hydrate of dicamphene, 116
- Hydrocarbons, unsaturated, 189
- Hydrochloric acid, electrolysis of, 213
- Hydrocinchonine, 117
- Hydrocinchonamide, 139
- Hydrogen and bromine, effect of light on combination of, 111
and carbon, determination of, 215
estimation of, 140
boiling point and density of liquid, 261, 282
bromide, action of in presence of ether on carbohydrates, 282
chloride, drying of, 198
liquid, 227
liquefaction of, 216, 237
nascent, 180
nitrogen, and carbon, values of atomic weights of, 224
peroxide on carbohydrates, 233
refractivity of, 1
- Hydronic acid, 181
- Hydroquinones and quinones, 11
- Hydroxylamines, action of on imid-chlorides, and anilidoximes, 235
- Hydroxylamine, estimating, 132
- Hygiene and Demography International Congress of, 72, 106
- Hyposulphite, decomposition of, 95
- Hysterical chemistry, 3, 16
- IMBERT, H., action of cyanamide on bromanile, 116
and A. Anstruc, neutralisation of glycerophosphoric acid by the alkalis, 36
and G. Belugou, heat of neutralisation of glycerophosphoric acid, 36
and J. Pages, estimation of the phosphoglycerates, 298
- Imidchlorides, action of hydroxylamines on, 225
- Incandescent bodies, investigation of, 249
- Indian and American podophyllum, 113
- d, estuff, Delphinium salil, colouring matters of, 126
- Indicators, 142
lecture experiments with, 131
- Inorganic compounds, constitution of, 213
- Institute of Chemistry, 46
- "Institution of Mining and Metallurgy, Transactions of" (review), 23
- Institution, Royal, 24, 82, 96, 115, 141, 166, 211, 223, 250, 285
- International Congress of Applied Chemistry, 24, 214
- Hygiene and Demography, 72, 106
- Health Exposition, 83
- Photographic Exhibition, 87
- "Introduction to the Study of Organic Chemistry" (review), 273
- Iodide of glucinum, 266
of tungsten, 204
- Iodine, bromine, and chlorine, separation and estimation of, 67
on antipyrine, 141
preparation of pure, 56
separation and estimation of, 276
- Iron and manganese, separation of, 201
and steel, determination of chromium in, 187
carbon in, 105
gold deposited on, 129
pipes, lining, 100
- Isocyanic ethers, 59
- Isomeric bornylamines, 197
changes in organic compounds, generalisation of, 9
- Isomers in the piperidine series, 225
- Isonitramine and nitroso-isobutyric acid, 117
and nitramine and their ethers, 165
fatty acids, etherification of, 117
- Isoquinolein and tetrahydroisoquinolein, 224
- Itzig, H. and A. Roenheim, manganese molybdates, 105
- JACK, O., and A. Claus., chlorine and bromine derivatives of naphthylamines, 128
- Jarry, M., ammoniacal bromides of silver, 261
- Jean, F., analysis of raw sulphide of sodium, 153
separation of lead, copper, and arsenic, 172
- Jervis, H., laboratory notes, 5, 133
- Joannis, A., existence of a cuprous sulphate, 23
- Job, A., new compounds of cerite metals, 75
- Johnson, A. E., "The Analyst's Laboratory Companion" (review), 104
- H., hydrolysis of starch, 208
- Jorgensen, S. M., constitution of cobalt, nickel, and rhodium bases, 213
- Jolly, L., researches on organic phosphorus, 116
- Jones, A. W., illustration of the law of multiples, 210
H. O., and S. H. King, dissociation of electrolytes, 243
atomic weight of praseodymium and of neodymium, 280, 292
boiling point apparatus, 59
- H. J., "The Freezing-point, Boiling-point, and Conductivity Methods" (review), 212
- L. C., action of carbon dioxide on soluble borates, 294
- Judson, W., and J. W. Walker, reduction of bromic acid and law of mass action, 149
- Julliard, M., preparation of distilled waters, 12
- Junghahn, A., preparing tetrazin derivatives, 226
- KAEPPPEL, F., estimation of manganese, 201
- Kahl, L., and O. Möhlan, formaldehydtrioxyfluorondicarboxylic acid, 225
products of action of formaldehyd on gallic acid, 225
- Kastle, J. H., and W. A. Beatty, effect of light on combination of hydrogen and bromine, 111
- Kekulé memorial, 88, 285
- Kern, S., metallurgical notes, 192
production of steel castings, 86
- Ketone acids, ferric chloride on etheral salts of, 149
- Ketones, brominated, 166
new cyclic, 35
- King, S. H., and H. C. Jones, dissociation of electrolytes, 243
- Kipping, F. S., and W. J. Pope, separation of optical isomers, 211
- Koetschet, J., and P. Monnet, saccharines, 96
- Kopfer's method for determination of carbon and hydrogen, 215
- Krypton, 270
- Ktypeite, a new form of carbonate of lime, 140
- Kunkell, F., and W. Scheven, brominated ketones, 166
- Kunz, G. F., "Production of Precious Stones in the United States in 1896" (review), 44
- Kurloff, B., equilibrium between ammonium nitrate and ammonia, 189
- Kuspert, F., and K. A. Hofmann, estimating hydroxylamine and hydrazine, 132
- LABORATORY, improved superheater for, 25
notes, 5, 133
- "Laboratory Experiments on the Glass Reactions, and Identification of Organic Substances" (review), 296
- Lacroix, A., anhydrous sulphate of lime produced by dehydration of gypsum, 83
formation of anhydride, 116
- ktypeite, a new form of carbonate of lime, 140
- Ladd, E. F., "A Manual of Quantitative Chemical Analysis" (review), 272
- Ladenburg, A., ethylpiperidine and its methyl derivative, 225
isomers in the piperidine series, 225
n-methylpiperoline, 226
- Landauer, J., "Spectrum Analysis" (review), 139
- Lander, G. D., and T. Purdie, action of alkyl iodides on silver malate and on silver lactate, 163
- Landolt, Dr. H., "Das Optische Drehungsvermögen Organischer Substanzen und dessen Praktische Anwendungen" (review), 284
- Lapworth, A., generalisation of isomeric changes in organic compounds, 9
sulphonation of benzophenone and diphenylmethane, 210
- Lauder, A., and J. J. Dobbie, Kopfer's method for determination of carbon and hydrogen, 215
- Laurent, J., absorption of organic matter by roots, 11
- Laurolic acid, 210
experiments on, 198
- "Lead and Copper Smelting and Copper Converting, Notes on" (review), 43
- Lead and potassium, double iodide of, 191
copper, and arsenic, separation of, 172
free ammonium salts of tartaric and citric acids, 119
salt, a curious, 191
- Lean, B., and W. H. Whatmough, preparation of pure iodine, 56
- W., and J. N. Collie, production of some chloropyridine carbonylic acids, 285
- Lebeau, P., treatment of the emerald by the electric furnace, 285
- P., anhydrous fluoride of glucinum and oxyfluoride of glucinum, 288
borocarbide of glucinum, 289
iodide of glucinum, 266
preparation of alloys of glucinum, 44
glucinum by electrolysis, 172
- Leclère, A., analysis of silicates, 27
- Lecture experiments, 102
with indicators, 131
- Leduc, A., composition of air and density of gases, 95
dissociation and polymerisation of gases and vapours, 23
mixture of gases, 71
- Lees, F. H., and W. H. Perkin, aluminium chloride on camphoric anhydride, 210
- Leffler, R. L., estimation of chromium, 156
separation of aluminium, 265
- Legrand, E., electric conductivity of solutions of permanganate of potash, 224
- Lehfeldt, R. A., properties of liquid mixtures, 128
- Leleux and Gin, M. M., dissociation of carbides of barium and manganese, 165
- Lemoult, P., chlorocyanamide, 10
isocyanic ethers, 59
silver cyanamide, 12
- Leonard, N., atomic weight of boron, 104
- Lépine, E., urobiline and biliary pigments, 11
- Le Roy, F., electrical resistance of crystallised silicon, 71
- Lerze, F., and W. Will, nitration of carbohydrates, 152
- Levy, A., and F. Marboutin, estimation of oxygen dissolved in water, 237
- Ley, H., action of hydroxylamines on imidchlorides and anilidoximes, 225
- Léys, A., detection of rocou in milk, 262
- Lifschütz, J., decomposition of wool fat, 165
- Light, chemical influence of, 248
effect of on combination of hydrogen and bromine, 111
- Lime and sulphuric acid, estimation of in waters, 135
phosphoglycerate of, 12

- Lindo-Gladding method of determining potash**, 263, 275
Liquid air by the gallon, 113
 hydrogen, 227
 boiling point and density of, 282
 mixtures, properties of, 128
Liquids, sterilisation of, 188
 temperature on rotatory power of, 10
Liversidge, A., corrosion of aluminium, 207
 crystallised carbon dioxide, 216
Ljubarsky, E., liquid fatty acids of fat of dogfish, 129
J., and A. Bogorodsky, allyl-ethylphenylcarbinol, 129
Lloyd, L. L., and J. J. Sudborough, stereochemistry of unsaturated compounds, 7
Lbb, W., and A. Claassen, "Quantitative Chemical Analysis by Electrolysis" (review), 176
Lodge, O., electric signalling without connecting wires, 58
London, City and Guilds of, Institute, 24, 72
 water supply, 33, 90, 122, 185, 232, 278
Lossen, W., bromsuccinic acids, 116
Lowenstein, H., and T. H. Norton, salts of dinitro-alpha-naphthol, 90
Lowry, T. M., stereoisomeric derivatives of camphor, 259
Lugan and Dutremblay, M.M., preparation of oxygen, 11
Lumiere and Seyewitz, M.M., reaction of aldehyds and acetones, 236
Lumsden, J. S., and J. Walker, determination of molecular weights, 236
Lunge, G., carbonic acid in bicarbonates, 109
 and **E. Harbeck**, carbon monoxide on platinum and palladium, 103
 estimating carbon in iron, 105
 separation of ethylene and benzene vapour, 105
Lipke, Dr. R., "The Elements of Electro-Chemistry, treated Experimentally" (review), 248
Lupton, S., "Notes on Observations" (review), 247
- MACHADO, V.**, novel facts in Crookes tubes, 23
Madan, H. G., properties of methylene di-iodide, 199
Magnesium and solution of copper sulphate, interaction of, 127, 139
 basic salts of, 83
 in qualitative analysis, 105
Mahon, R. W., phosphorus in steel, 117
Makin, C. J. S., composition of Atlantic Ocean, 135, 171
Malosse, T., and J. Battandier, retamine, 11
Manganese and iron, separation of, 201
 bronze, 192
 estimation of, 201, 242, 255
 in plants and vegetable soils, 108
 molybdates, 105
 precipitated by the chlorate process, condition of oxidation of, 269, 278
 separation of from chromic acid, 131
Manganic salts, 125
"Manual of Quantitative Chemical Analysis" (review), 272
Marboutin, F., and A. Levy, estimation of oxygen dissolved in water, 237
 and **M. Molinié**, volumetric estimation of combined sulphuric acid, 46
Markownikoff, W., para-pseudo-propyl-naphthene acid, 189
- Maraden, F., and J. J. Dobbie**, o-chlorobrombenzene, 103
 Mass action, law of, 149
Matches, manufacture of, 286
Mather, T., and W. E. Ayrton, galvanometers, 246
Matignon, O., carbide of sodium 35
 preparation of carbide of sodium and monosodic acetylene, 177
 sodium on acetylene, 177
Matthews, F. E., benzene hexabromide, 125
 derivatives of benzophenone, 198
McCrae, J., and P. Frankland, position isomerism and optical activity—rotatory powers of diethylic monobenzoyl and monotoluyil tartrates, 162
McGill University, Montreal, 12
 Meal, determining the fineness of, 249
Meker, G., and A. Etard, crystallised hydride of dicamphene, 116
Méker, G., new method for attack of platinum, 19
Melikoff, P., and L. Pissarjewsky, ammonium peroxide, 165
 Melted salts, dissociation spectra of, 88
Mercury and bismuth salts, separation of, 165
 and concentrated sulphuric acid, reactions between, 40
 sulphuric acid on, 136
Metals, analysis of samples of, 121
 Metallic phosphides, 227
 salts of natural yellow colouring matters, 126
 sulphides as electrodes, 189
Metallurgical notes, 192
 Methylcyclohexenone, 59
 Methyl-diethylethylene, 129
 Methylene di-iodide, properties of, 199
 Methylene dimonochloracetyl tartrates, rotation of, 80
 Methyl glycerates, effect of mono-, di-, and tri-chloracetyl groups on rotatory power of, 80
 Methyl iodide and potash on nitrosophenol, 226
 tartrates, optical rotations of, 163
"Metric System of Weights and Measures, an Elementary Treatise on" (review), 187
Milk, coagulating-point of, 83
 detection of rocou in, 262
 sugar and grape-sugar, estimation, 147
 estimation of casein in, 106
Miller, F. W., and O. Baskerville, decomposition of sulphuric acid, 191
 reactions between mercury and concentrated sulphuric acid, 40
Mineral matter in rubber goods, 74
Minerals from Staassfurth potash beds, analysis of, 121
Minium, assay of, 250
Möhlau, R., and L. Kahl, formaldehyd-trioxyfluorondicarboxylic acid, 225
 products of action of formaldehyd on gallic acid, 225
Moissan, H., formation of alkaline carbides, alkaline-earth carbides, and carbide of magnesium, 85
 method of preparing carbides, 11
 and **M. Deslandres**, spectral research on atmospheric air, 288
"Molecular Asymmetry, Researches on" (review), 70
Molecular weight of solid bodies, 165
 weights, determination of, 236
- Molecular weights of permanganates, perchlorates, and periodates in solution**, 236
Molecules and liquefaction heats, 35
Molinié, M., and F. Marboutin, volumetric estimation of combined sulphuric acid, 46
Molybdi anhydride, reduction of, 45
Monazite, occurrence and extraction of, 134, 145
 sands, yttric earths in, 193, 204, 220, 229
Moncour, M., glycerophosphate of quinine, 298
Monnet, P., and J. Koetschet, saccharines, 96
Monochloracetyl groups, effect of on rotatory power of methylic and ethylic glycerates and tartrates, 80
Monotoluyil tartrates and diethylic monobenzoyl, comparative rotatory powers of, 162
Monomethylaniline, formation of, 57
Monosodic acetylene, preparation of, 177
Montreal, McGill University, 12
Moreau, M., and P. Cazeneuve, aromatic diurethanes and piperazine, 45
 urethanes of concine, 116
 piperidine on carbonic ethers from the phenols, 36
Morgan, G. T., action of formaldehyds on amines of the naphthalene series, 258
Morner, K. A. H., estimation of carbon and hydrogen, 140
Morphine, nitrogen-free decomposition products of, 152
Morrill, R. S., and J. M. Crofts, emolic and ketonic forms of ethylic acetoacetate, 235
 ferric chloride on the ethereal salts of ketone acids, 149
Morton, H., liquid air by the gallon, 113
Moths, destruction of, 47
Mourlo, J. R., decomposition of hyposulphite of strontium, 95
 properties of phosphorescent sulphide of strontium, 201
 duration of phosphorescent power of sulphide of strontium, 56
Mourlot, A., anhydrous sulphide of barium, 140
Mulliken, S. F., and A. A. Noyes, "Laboratory Experiments on the Class Reactions and Identification of Organic Substances" (review), 296
 Multiples, illustration of the law of, 210
Mutteleit, F., chloride of benzoyl-p-nitrated on mono-substituted ortho-diamines, 96
Mylus, A., oxycobaltamines and anhydrocobaltamines, 201
- NAPHTHINDOL derivatives**, synthesis of, 225
 Naphthol ether, oxides of, 188
 Naphthoquinolines, 188
 Naphthylamines, chlorine and bromine derivatives of, 128
Nardin, L., and E. Bourquelot, preparation of gentianose, 71, 271
Nascent hydrogen, 83, 180
Neodymium, 161, 193
 atomic weight of, 280, 292
 spectrum and nature of, 219
Newton's rings, method of viewing, 199
New York Agricultural Experiment Station, 213
Nickel, constitution of, 213
Nieloux, M., carbonic acid in air, 165
 and **A. Desgrez**, decomposition of chloroform, 165
- Nitramine and isonitramine, and their ethers**, 165
Nitrate of mercury and trimethylcarbinol, combination obtained with, 225
Nitric oxide, absorption of by ferrous salts, 268, 290
Nitrification of soils, 18
Nitrified bodies, action of oxidants on, 225
Nitro-compounds in presence of free nitrogen, 188
 products of explosive decomposition of, 152
Nitro- and amido-oxyntidines, production of, 124, 125
Nitrogen and organic acids, 164
 the aldehyds, 164
 carbon, and hydrogen, values of atomic weights of, 224
 chloride, end-product of action of on dimethylaniline, 225
 compounds, synthesis of, 117
 refractivity of, 1
Nitroso-isobutyric acid and isonitramine, 117
Nitrosophenol, diazomethane and methyl iodide and potash on, 226
N-methylpipercoline, 226
Non-conducting compounds, spectral analysis of, 218
Normantine, 72
Norton, T. H., and H. Lowenstein, salts of dinitro-alpha-naphthol, 90
"Notes on Observations" (review), 247
Nottingham University College, 46
Noyes, W. A., "Organic Chemistry for the Laboratory" (review), 212
 camphor and its derivatives, 70
A. A., and S. P. Mulliken, "Laboratory Experiments on the Class Reactions, and Identification of Organic Substances" (review), 296
- OBITUARY, Lord Playfair**, 261
O'Connor, H., "Gas Engineer's Pocket Book" (review), 224
Oil, estimation of rosin and rosin oil in linseed, 287
 of acetone, manufacture of, 83
Oleic acid, constitution of, 259
Ontario, corundum deposits in, 142
"Optical Activity of Organic Substances and its Applications" (review), 284
Optical isomerides, separation of, 211
 phenomena, dynamical illustrations of, 128
Optimus camera, 207
Orcinol, condensation of chloral hydrate with, 207
Organic acids and nitrogen, 164
 bases, combination with oxygenated salts, 213
 bodies and electrolytes, determinations of dielectric constants of, 13, 25, 37
 compounds, chemical action exercised by electric discharge on, 140
 matter, absorption of by roots, 11
 phosphorus, researches on, 116
 substances, combustion of in aqueous solution, 213
"Organic Chemistry for the Laboratory" (review), 212
"Organic Chemistry, Introduction to the Study of" (review), 273
Osborne, T. B., chemical nature of diastase, 225
Osmotic pressure and cryoscopy, 174
 and electrolytic dissociation, 165

Onabaine, products from splitting up by hydrolysis, 285
 researches on, 83
 Oxalic acid, influence of altitude and warmth on decomposition of by sunlight, 11
 Oxidants, action of on nitrified bodies, 225
 Oxides of cerium and thorium, separation of, 245, 254
 Oxyacannabin from Indian hemp, 114
 Oxybaltamines and anhydroxycobaltamines, 201
 Oxyfluoride of glucinum, 288
 Oxygen, absorption of by pyrogallate of potash, 249
 action of on sulphide of carbon, 248
 dissolved in water, estimation of, 237
 on dissolved formaldehyd, 45
 preparation of, 11
 refractivity of, 1
 Oxygenated salts, combination of organic bases with, 213
 Oxyptomaine, 140
 Oxytriazoles, formation of, 162
 Ozonator, 274, 286

PAGES, J., and H. Imbert, estimation of the phosphoglycerates, 298
 Palladous chloride, carbonic oxide on, 140
 Palmaer, W., working of dropping electrodes, 270
 Panting, L. C., and J. Wade, preparation of anhydrous hydrogen cyanide and carbon monoxide, 124
 "Paper-makers, Directory of," 166
 Para-acetate of phenyl, preparation of, 236
 Paranitrobenzaldehydorthoethylphonic acid, oxidation of paranitrotoluenesulphonic acid to, 236
 Paranitrotoluenesulphonic acid, oxidation of to dinitroethylbenedisulphonic acid and to paranitrobenzaldehydorthoethylphonic acid, 236
 Para-pseudo-propylnaphtheneic acid, 189
 Paris Exhibition of 1900, 285
 Pasteur, L., "Researches on Molecular Asymmetry" (review), 70
 Patein, G., compounds of antipyrin with the aldehyde, 47, 95
 Patterson, T. S., and P. Frankland, effect of mono-, di-, and tri-chloracetyl groups on rotatory power of methylic and ethylic glycerates and tartrates, 80
 Pawlowski, B., fluorescent substance, 226
 Payne, G. F., "Commercial Fertilisers and Chemicals" (review), 211
 Peachey, S. J., and W. J. Pope, resolution of tetrahydropapaverine into its optically active components, 235
 Peak of Tenerife, 103
 Pechmann, H. V., and E. Scel, diacetomethane and methyl iodide and potash on nitrophenol, 226
 and W. Schmitz, diacetomethane on aromatic nitroso-bases, 226
 Percarbonate of potassium, 65
 Perchlorates in solution, molecular weight of, 236
 of the alkalis and the alkaline earths, formation of, 152
 Periodates in solution, molecular weight of, 236
 Perkin, A. G., yellow colouring matter of leaves of *Arctostaphylos uva ursi*, 208

Perkin, A. G., and J. A. Pilgrim, colouring matter of Indian dye-stuff, Delphinium zalli, 126
 and F. J. Wood, metallic salts of natural yellow colouring matters, 126
 yellow colouring matters of adulterants of Sicilian saffron, 208
 and R. W. Collinson, lauronic acid, 210
 and E. W. Haworth, condensation of formaldehyd with ethylic malonate, 114
 and F. H. Lees, aluminium chloride on camphoric anhydride, 210
 and C. H. G. Sprinkling, bromoacetal on the sodium derivatives of ethylic malonate, 210
 and J. F. Thorpe, synthesis of cis- and trans-canoic acid, 209
 Perman, E. P., rate of escape of ammonia from aqueous solution, 79
 Permanganate of potash, electric conductivity of solutions of, 224
 Permanganates in solution, molecular weight of, 236
 Perot, A., and C. Fabry, interferential spectroscopy, 82
 Persulphuric acid, 39
 Pervanadic acid, 201
 "Phase Rule" (review), 34
 Phelps, I. K., combustion of organic substances in aqueous solution, 213
 Phenol-glyoxylic acids, 46
 Phenylenediamine, diazo-compounds of, 166
 Phenylhydrazin, action of ethylic aldehyd on, 236
 coloured reaction of, 116
 estimation of, 232
 Philadelphica Museums, 72
 Phosphoglycerate of lime, 12
 Phosphoglycerates, acid, 285
 estimation of, 141, 237, 298
 Phosphorescent strontic sulphide, production of, 95
 sulphide of strontium, properties of, 201
 Phosphoric acid determination, 32
 Gladding's method for, 242
 reaction of on glycerin, 237
 anhydride with benzene, combination of, 123
 di-ethers, 285
 mono-ethers, 274
 Phosphorous oxide, 20
 Phosphorus, 88
 in steel, 117
 "Photographs, Chemistry for" (review), 44
 Photographic Exhibition, International, 87
 plate, action of certain metals on, 167
 Society, Royal, 47
 Phthalic green, constitution of, 44
 "Physical Chemistry, Text-book of" (review), 247
 Physical Society, 52, 58, 81, 103, 128, 150, 199, 246, 260, 283
 Pichard, P., manganese in plants and vegetable soils, 108
 Pilgrim, J. A., and A. G. Perkin, colouring matters of Indian dye-stuff, Delphinium zalli, 126
 Pinkus, G., action of benzhydrazide on glucose, 152
 Piperazine, 45
 Piperidine on carbonic ethers from the phenols, 36
 series, isomers in, 225
 Pissarjewski, L., and P. Melikoff, ammonium peroxide, 165
 Pitman, J. R., sulphuric acid on mercury, 136
 Plants and vegetable soils, manganese in, 108

Plaster-of-Paris method in blow-pipe analysis, 15
 Platinichlorhydric acid, behaviour of some salts of, 203
 Platinichloride, dissociation of, 79
 Platinum, method for attack of, 19
 monochloride, production of, 79
 Playfair, Lord, obituary, 261
 Podophyllum, Indian and American, 113
 Polymorphism of fluorite, 116
 Poole, H., "The Caloric Power of Fuels" (review), 176
 Pope, F. G., and J. T. Hewitt, derivatives of bromotolylhydrazine, 56
 W. J., and F. S. Kipping, separation of optical isomerides, 211
 and S. J. Peachey, resolution of tetrahydropapaverine into its optically active components, 235
 Porter, T. C., method of viewing Newton's rings, 199
 new theory of geyser action, 103
 Peak of Tenerife, 103
 volatility of sulphur, 150
 Position-isomerism and optical activity, 162
 Potain and Drouin, MM., chloride of palladium for detection of carbonic oxide in the air, and conversion of this gas into carbonic acid, 212
 Potash, Lindo-Gladding method of determining, 263, 275
 Potassium and lead, double iodide of, 191
 and rubidium, fluosilicate and fluophosphate of, 165
 carbonate, 126
 Pouget, M., alkaline sulphantimonites, 274
 Praseodidymium and neodidymium, 161
 Praseodymium, atomic weight of, 280, 292
 "Precious Stones, Production of in the United States in 1896" (review), 44
 Prescott, A. B., alkyl bismuth iodides and bismuth iodides of vegetable bases, 138
 Prismatic spectra, method of reducing, 260
 "Proceedings of the Chemical and Metallurgical Society of South Africa," Vol. I. (review), 248
 Proteid precipitates, contributions to the chemistry of, 249
 Protoside of chromium and carbonate of soda, 177
 Purdie, T., and G. D. Lander, action of alkyl iodides on silver malate and on silver lactate, 163
 Pyridine, action of chlorine on, 236
 behaviour of with propionic, acetic, and formic acids during distillation, 45
 chlorine derivatives of, 210
 combinations of with formic and acetic acids, 249
 Pyrocatechin, new derivatives of, 106
 Pyrogallate of potash, absorption of oxygen by, 249

QUALITATIVE analysis, sodium, magnesium, and aluminium in, 105
 Quantitative analysis, sodium peroxide in, 123
 "Quantitative Chemical Analysis" (review), 104, 272
 "Quantitative Chemical Analysis by Electrolysis" (review), 176
 Quinine, glycerophosphates of, 297, 298

Quinoleic bases, 212
 Quinones and hydroquinones, 11
 of high molecular weights, heat of formation of, 262
 Quinoximes, 285

RAMAGE, H., and W. N. Hartley, analysis of samples of metals, of chemical preparations, and minerals from Stassfurth potash beds, 121
 Ramsay, W., the new gas, 297
 and M. W. Travers, fergusonite, 64
 homogeneity of helium, 61
 new constituents of atmospheric air, 287
 refractivities of air, oxygen, nitrogen, argon, hydrogen, and helium, 1
 Rays emitted by compounds of uranium and of thorium, 249
 Reocour, A., chromosulphochromic acids, 45
 Reychler, A., osmotic pressure and cryoscopy, 174
 sulphonic derivatives of camphor, 177
 Reynolds, W. E., chemical properties of concentrated solutions of certain salts, 126
 Rhodium bases, constitution of, 213
 Rice, C. E., manganic salts, 125
 Richards, T. W., relation of taste of acids to their degree of dissociation, 91
 and G. P. Baxter, revision of atomic weight of cobalt, 20, 30
 Riehm, H. D., the new gases, 297
 Riegler, E., estimating grape sugar and milk sugar, 141
 Roberts-Jones, M., "Handbook to the Workmen's Compensation Act, 1897" (review), 44
 Robin, L., estimation of sulphuric acid and lime in waters, 135
 Rocou, detection of in milk, 262
 Rodger, J. W., and J. S. S. Brame, optical rotations of methyl and ethyl tartrates, 163
 Rohland, P., behaviour of some salts of platinichlorhydric acid, 203
 Röhrner, H., condensation products of furolic acids and furoacroleins, 225
 Roots, absorption of organic matter by, 11
 Rosenheim, A., and H. Itzig, manganese molybdates, 105
 O., and P. Schidrowitz, Fehling's solution, 97
 Rosin and rosin oil, estimation of in linseed oil, 287
 Royal Institution, 24, 82, 96, 115, 141, 166, 211, 223, 250, 285
 Photographic Society, 47
 Society, 223, 272
 Rubber goods, mineral matter in, 74
 Rubidium and potassium, fluosilicate and fluophosphate of, 165
 Ruhemann, S., formation of dihydroxypyridine, 162
 and K. E. Browning, formation of ethylic dihydroxydinicotinate from ethylic cyanacetate, 115
 Russell, W. J., action of certain metals on a photographic plate, 167

SACCHARINES, 96

Saglio, M., enamels of high dilatation, 272
 Sagnac, G., transformation of the X rays of metals, 23
 Salicylic acid in foods, 136

- Salt solutions, dissociation in mixed, 189
- Salts, chemical properties of concentrated solutions of certain, 126
- dissociation spectra of melted, 28
- of dinitro- α -naphthol, 90
- Sandstrom off West coast of Africa, 119
- Sanitary Institute, 47, 214
- Saytzeff, A., methylthylethylene, 129
- and A. Tscherbakoff, action of sulphuric acid on elaidic acid, 129
- Seel, E., and H. von Pechmann, diazomethane and methyl iodide and potash on nitrophenol, 226
- Schenck, R., investigation of crystalline liquids, 286
- Scheuer, A., pervanic acid, 201
- Scheven, W. and F. Kunczell, brominated ketones, 166
- Schidrowitz, P., and O. Rosenheim, Fehling's solution, 97
- Schiff reaction applied to some substituted fuchsin, 83
- Schjerning, H., contributions to the chemistry of proteid precipitates, 249
- Schlagdenhaufen, M., imprints in raw copper, 72
- Schlossing, T., nitrification of soils, 18
- Schmitz, W., and H. Pechmann, diazomethane on aromatic nitroso-bases, 226
- Schranz, W., and P. Dobriner, analysis of caustic soda, 229
- analysis of sulphide and sulphhydrate of sodium, 108
- Schryver, S. B., experiments on lauronic acid, 198
- Schutzenberger, F., and O. Boudouard, yttric earths in monazite sands, 193, 204, 220, 229
- Scorification versus cupellation, 39
- Sell, W. J., and F. W. Dootson, chlorine derivatives of pyridine, 210
- action of chlorine on pyridine, 236
- Sewage, septic treatment of, 270
- Seyewitz and Lumière, MM., reaction of aldehyds and acetones, 236
- Shenstone, W. A., and W. T. Evans, influence of discharge of electricity on atmospheric air, 102
- Shorey, E. C., principal smide of sugar-cane, 75
- Sicilian sumach, yellow colouring matters of adulterants of, 208
- Sielaff, H., etherification of isonitramine fatty acids, 117
- Silicates, analysis of, 27
- Silicic ether, a natural, 214
- Silicide of chromium, 188
- Silicon, electrical resistance of, 71
- line spectrum of, 206
- refraction and dispersive power of, 271
- Silver, ammoniacal bromides of, 261
- and gold, melting points of, 115
- cyanamide, 12
- lactate, action of alkyl iodides on, 163
- malate, action of alkyl iodides on, 163
- Simcock, A., and O. Hinsberg, synthesis of naphthindole derivatives, 225
- Simon, L., coloured reaction of ordinary aldehyd, 29
- reaction of phenylhydrazine, 116
- Skinner, S., affinity constants of dihydroxymaleic, dihydroxyfumaric, dihydroxytartaric, and tartaric acids, 235
- Smith, C., O. F. Cross, and E. J. Bevan, action of hydrogen peroxide on carbohydrates, 233
- carbohydrates of barley straw, 197
- C. E., testing of formaldehyd, 94, 98
- E. F., and D. S. Wallace, determination of cadmium, 93
- J. H., "An Elementary Treatise on the Metric System of Weights and Measures" (review), 187
- Smith, W. A., dissociation of dibasic organic acids, 274
- Smoke, determination of the weight of, 249
- Snape, H. L., di-isocyanides upon amido-compounds, 162
- Société d'Encouragement pour l'Industrie Nationale, 272
- Society, Chemical, 7, 54, 78, 100, 113, 124, 148, 160, 175, 197, 207, 233, 257, 282
- Physical, 52, 58, 81, 103, 128, 150, 199, 246, 260, 283
- Royal, 223, 272
- Royal Photographic, 47
- Soda, analysis of caustic, 229
- and protoxide of chromium, double carbonate of, 45
- Sodium derivative of ethylic malonate, bromacetal on, 210
- in qualitative analysis, 105
- on acetylene, 177
- peroxide in quantitative analysis, 123
- salt of malonic acid, addition of cyanogen to, 166
- thiosulphate, titration of, 213
- volumetric estimation of, 78
- Soehne, M., and C. F. Boehringer, ammonia test of Coccaium hydrochloricum, 183
- Soils, nitrification of, 18
- Solubility, heat of solution and degree of dissociation, relationship between, 189
- Solution, heat of, solubility and degree of dissociation, relationship between, 189
- Sonstadt, E., decomposition of auric chloride, 74
- dissociation of platinumchloride in dilute solution, and production of platinum monochloride, 79
- Spectral analysis of non-conducting compounds, 218
- research on atmospheric air, 288
- Spectroscope, an interferential, 82
- "Spectrum Analysis, by John Landauer" (review), 139
- Spectrum of benzene, absorption bands in, 103
- of cadmium in vacuo, 71
- Sperber, Dr. J., chemically inactive elements, 87
- Speyers, O. L., "Text-book of Physical Chemistry" (review), 247
- Spivey, W. T. N., T. H. Easterfield, and T. B. Wood, cannabinol, 150
- Sprankling, C. H. G., and W. H. Perkin, bromacetal on the sodium derivative of ethylic malonate, 210
- Stannous salts, titration of, 153
- Stansfield, A., thermo-electric pyrometers, 151
- Starch, hydrolysis of, 208
- in flour, estimation of, 107
- Stassfurth potash-beds, analysis of minerals from, 121
- Steel and Iron, determination of chromium in, 187
- castings, production of, 86
- phosphorus in, 117
- Stein, S., "Sugar, Home-grown and Home-manufactured" (review), 187
- Stereochemistry of unsaturated compounds, 7
- Stockholm tar, acetyl-furfuran in, 45
- Stockwell, B. M., and G. Young, formation of oxtriazoles, 162
- Storer, F. H., "Agriculture in some of its Relations with Chemistry" (review), 163
- Sudborough, J. J., and M. E. Feilman, formation and hydrolysis of esters, 7
- and L. L. Lloyd, stereochemistry of unsaturated compounds, 7
- Sugar-cane, principal amide of, 75
- Sugar, estimating grape and milk, 141
- "Sugar, Home-grown and Home-manufactured" (review), 187
- Sulphate of copper, interaction of magnesium and solution of, 139
- of quinine, commercial, 298
- Sulphide, analysis of, 108
- of carbon, action of oxygen on, 248
- of sodium, analysis of raw, 153
- of strontium, duration of phosphorescent power of, 36
- decomposition of, 95
- Sulphocyanic ethers, decomposition of, 188
- Sulphonie derivatives of camphor, 177
- Sulphur, 88
- dioxide, apparatus for determining composition of, 213
- volatility of, 150
- Sulphuric acid, action of on elaidic acid, 129
- and lime, estimation of in waters, 135
- decomposition of, 191
- on mercury, 136
- reactions between mercury and, 40
- volumetric estimation of, 46
- Sulphhydrate of sodium, analysis of, 108
- Superheater for laboratory purposes, 25
- Swinton, A. A. C., circulation of a gaseous matter in a Crookes tube, 150, 260
- "TABLES for Qualitative Chemical Analysis" (review), 207
- Takamine, J., diastatic fungi, 137
- testing diastatic substances, 173
- Tanret, C., nitrate, sulphate, chlorate, and phosphate of ammonia on *Aspergillus niger*, 45
- Tartrates, effect of mono-, di-, and tri-chloracetyl groups on rotatory power of, 80
- new ferment from, 106
- Tartaric acid, affinity constants of, 235
- Tassilly, M., basic salts of magnesium, 83
- Telle, F., estimation of combined sulphuric acid, 141
- Temperature on chemical reactions, 274
- Teneriffe, Peak of, 103
- Terebic acid, synthesis of, 83, 286
- Terpenes, researches on the, 57
- Terrat, P., testing for diastase from barley, 12
- Tetrahydroisoquinoline and isoquinoline, 224
- Tetrahydropapaverine, resolution of into its optically active components, 235
- Tetramethyl-diamido benzophenone, some derivatives of, 273
- Tetrazin derivatives, preparing, 226
- "Text-book of Physical Chemistry" (review), 247
- Theobromine, solubility of in aqueous solutions of salts, 153
- Thermo-chemical theory of a battery with carbon electrodes, 83
- Thermo-effects produced under influence of high pressure, 192
- Thermo-electric pyrometers, 151
- Thermophones, 192
- Thimm, C. A., "French Self-taught, with Phonetic Pronunciation" (review), 296
- Thiosulphates, interaction of cyanides with, 131
- Thiourea derivatives, 165
- Thomas, V., absorption of nitric oxide by ferrous salts, 290
- chloridising action of ferric chloride in the aromatic series, 285
- Thomas, M., absorption of nitric oxide by ferrous salts, 268
- Thompson, S. P., method of viewing Newton's rings, 199
- Thoms, H., choline and trigonelline in seeds of *Strophanthus*, and preparation of strophanthine, 225
- Thomsen, J., conical group of inactive elements, 120
- separation of helium from a natural compound, 190
- Thorite, occurrence and extraction of, 134, 145
- Thorium and cerium, separation of oxides of, 245, 254
- atomic weight of, 160
- chemistry of, 160
- rays emitted by compounds of, 249
- separation of, 97
- Thorpe, J. F., and W. H. Perkin, synthesis of cis- and trans-carbonic acid, 209
- Tickle, T., and J. N. Collie, production of some nitro- and amido-oxylindines, 124
- Tingle, J. B., "Spectrum Analysis, by John Landauer" (review), 139
- Tommasi, D., nascent hydrogen, 83, 180
- thermochemical theory of a battery with carbon electrodes, 83
- Townsend, C. F., "Chemistry for Photographers" (review), 44
- Trans- and cis-carbonic acid, synthesis of, 209
- Traube, J., molecular weight of solid bodies, 165
- osmotic pressure and electrolytic dissociation, 165
- W., addition of cyanogen to sodium salt of malonic acid, 166
- hydrazinacetic acids, 166
- synthesis of nitrogen compounds, 117
- Travers, M. W., and W. Ramsay, fergusonite, 64
- homogeneity of helium, 61
- new constituent of atmospheric air, 287
- refractivities of air, oxygen, nitrogen, argon, hydrogen, and helium, 1
- Traubert, F., and L. Varino, separation of mercury from bismuth salts, 165
- Trichloracetyl groups, effect of on rotatory power of methylic and ethylic glycerates and tartrates, 80
- Trigonelline and chlorine in seeds of *strophanthus*, and preparation of strophanthine, 225

- Trillat and Adrian, M.M., acid phosphoglycerates, 285
estimation of phosphoglycerates, 141, 237
phosphoglycerate of lime, 12
reaction of phosphoric acid on glycerin, 237
- Trimethylamine, combinations of with formic and acetic acids, 249
- Trimethylcarbinol and nitrate of mercury, combination obtained with, 225
- Trivalent alcohol of allyldipropylcarbinol, 129
- Trombeck, D., combination of organic bases with oxygenated salts, 213
- Truchot, P., occurrence and extraction of thorite, monazite, and zircon, 134, 145
- Tscherbakoff, A., and A. Saytzeff, action of sulphuric acid on elaidic acid, 129
- Tungsten, iodide of, 204
- Turnbull, A., and P. Frankland, rotation of ethylic and methylic dimonochloroacetyl-tartrates, 80
- "Tutorial Chemistry, Part II., Metals" (review), 200
- URANIUM**, rays emitted by compounds of, 249
- Urban, C., fractionation of yttria earths, 147
- Ureometer, a new water, 105
- Urobiline and biliary pigments, 11
- VALENTA**, E., and J. M. Eder, line spectrum of silicon, 206
- Valeur, A., heat of formation of some quinones of high molecular weights, 262
quinones and hydroquinones, 11
the quinonoximes, 285
- Vallot, M. and Mme., influence of altitude and warmth on decomposition of oxalic acid by sunlight, 11
- Van Laar, J. J., universal applicability of law of dilution, 189
- Van't Hoff, J. H., "The Arrangement of Atoms in Space" (review), 273
- Varino, L., and F. Treubert, separation of mercury and bismuth salts, 165
- Vedrdödi, V., determining the fineness of meal, 249
- Venable, F. P., revision of atomic weight of zirconium, 221, 231
- Verley, A., action of chloride of acetyl on acetate of phenyl, 236
- Verneuil, A., and G. Wyruboff, atomic weight of cerium, 45
separation of oxides of cerium and thorium, 245, 254
thorium and earths of cerite, 97
- Vongerichten, E., nitrogen-free decomposition products of morphine, 152
- Von Hansen, A., percarbonate of potassium, 65
- Von Hemptinne, A., decomposition of some substances under influence of electric vibration, 274
- Von Nimentowski, St., azimido-compounds of benzimidazole, 226
- WADDELL**, J., lecture experiments with indicators, 131
- Wade, J., "Introduction to the Study of Organic Chemistry" (review), 273
and L. O. Panting, preparation of anhydrous hydrogen cyanide and carbon monoxide, 124
- Walker, C. F., titration of sodium thiosulphate by iodic acid, 213
J., and J. S. Lumsden, determination of molecular weights, 236
and J. K. Wood, preparation of solid ammonium cyanate, 209
J. W., and W. Judson, reduction of bromic acid and law of mass action, 149
- Wallace, D. L., and K. F. Smith, determination of cadmium, 93
- Wallerant, F., polymorphism of fluorite, 116
- Walther, R., unsaturated hydrocarbons, 189
- Warren, H. N., scorification versus cupellation, 39
thermo-effects produced under influence of high pressure, 192
- Water, apparatus for determining composition of, 203
reservoirs of in soil, 117
supply, London, 33, 90, 122, 185, 232, 278
ureometer, 105
- Waters, estimation of sulphuric acid and lime in, 135
- "Weights and Measures, an Elementary Treatise on the Metric System of" (review), 187
- Weinland, R. F., and J. Alfa, fluosilicate and fluophosphate of potassium and rubidium, 165
- Wells, contamination of, 23
- Werner, A., constitution of inorganic compounds, 213
- Whitnough, W. H., and B. Lean, preparation of pure iodine, 56
- Wheeler, H. J., and A. L. Winton, Lindo-Gladding method of determining potash, 263, 275
- Wiesbaden Chemical Laboratory, 153
- Wilburgh's thermophones, 192
- Wildermann, M., determining freezing-points in very dilute solution, 8
- Will, W., and F. Lerze, nitration of carbohydrates, 152
- Willis, W. A., "The Workmen's Compensation Act, 1897" (review), 223
- Wilson, J. A., improved superheater for laboratory purposes, 25
- Winteler, F., formation of perchlorates of the alkalis and alkaline earths, 152
- Winter, J., congealing-point of milk, 83
- Winton, A. L., and H. J. Wheeler, Lindo-Gladding method of determining potash, 263, 275
- Wislicenus, W., copper compounds of dicarboxylglutamic ester, 165
- Wood, J. K., and J. Walker, preparation of solid ammonium cyanate, 209
- Wood, P. J., and A. G. Perkin, metallic salts of natural yellow colouring matters, 126
yellow colouring matters of adulterants of Sicilian saffron, 208
- T. B. W. T. N. Spivey, and T. H. Easterfield, cannabinalol, 150
- Wood of Goupia tomentosa, 114
products of distillation of, 142
- Wool fat, decomposition of, 165
- "Workmen's Compensation Act, 1897" (review), 44, 223
- Wyruboff, G., and A. Verneuil, atomic weight of cerium, 45
separation of oxides of cerium and thorium, 245, 254
thorium and earths of cerite, 97
- X RAYS** of metals, transformation of, 23
- YELLOW** azo-dye for colouring fats, detection of, 112
colouring matter of leaves of *Arctostaphylos uva ursi*, 208
matters, metallic salts of, 126
of adulterants of Sicilian saffron, 208
- Young, G., and E. Clark, ammonia and substituted ammonias on acetylurethane, 162
and B. M. Stockwell, formation oxytriazoles, 162
- S. W., titration of stannous salts, 153
- Yttria earths, fractionation of, 147
- Yttric earths in monazite sands, 193, 204, 220, 229
- Yvon, M. P., use of carbide of calcium for preparation of absolute alcohol, 52
- ZETTEL**, C., silicide of chromium, 188
- Zircon, occurrence and extraction of, 134, 145
- Zirconium, atomic weight of, 221, 231

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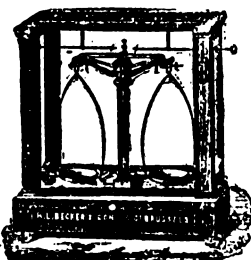
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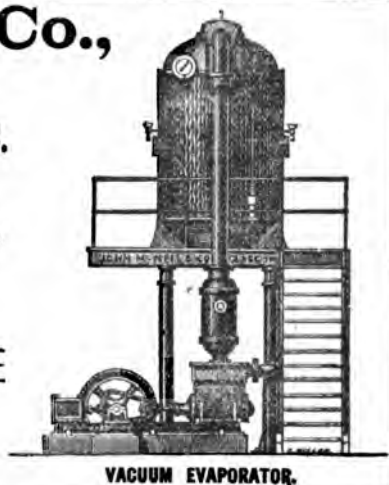
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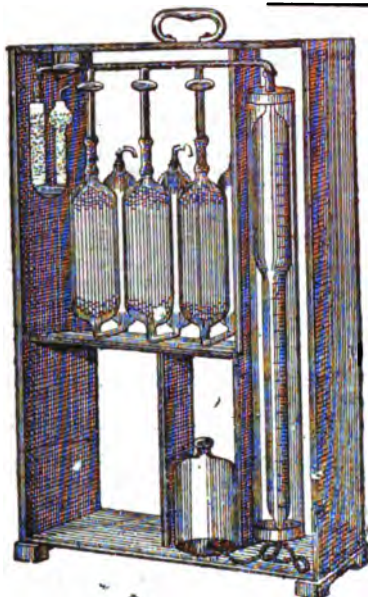
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